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PRMT SECTION

TECHNICAL REPORT

TRIAL BURN TEST RESULTS FOR MONSANTO CHEMICAL COMPANY
J. F. QUEENY PLANT
CAC INCINERATOR

Prepared by:
James A. Peters
Terran Corporation

Report No. TC-8903 Project No. QUE189

April 27, 1989

ABSTRACT

Distillation residues from the chloroacetyl chloride (CAC) process at Monsanto Chemical Company's J. F. Queeny plant in St. Louis, Missouri were burned at three test conditions representing Normal, Low and High waste feed rates as part of the RCRA trial burn for the CAC incinerator. Testing was conducted by PEI Associates, Inc. under the direction of Terran Corporation from January 31 to February 18, 1989. Results of the trial burn are reported for all influent/effluent streams -- CAC Residue feed, city water to quench and scrubber, quench effluent, scrubber effluent, and stack exhaust gas.

RCRA RECORDS CENTER

P.O. BOX 1410, FAIRBORN, OHIO 45324-1410 (513) 372-6345

April 28, 1989

Mr. Thomas C. Pauling
Environmental Engineer
Waste Management Program
Division of Environmental Quality
Missouri Department of Natural Resources
P.O. Box 176
Jefferson City, Missouri 65102

Re: Monsanto-Queeny CAC Incinerator Trial Burn Report

Dear Mr. Pauling:

Enclosed please find two (2) copies of the CAC Incinerator trial burn final report for the Monsanto Chemical Company J. F. Queeny plant in St. Louis, Missouri. Two (2) copies of the Appendices (Volumes I and II) are also enclosed.

If you have any questions or need additional information, please call me at 513/923-4774.

Sincerely,

James A. Peters

Environmental Consultant

cc: File

B. E. Huntsman - Terran

Mr. John J. Smith
USEPA Region VII
726 Minnesota Avenue
Kansas City, KS 66101 - 1 Copy

Enclosures

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1. SUMMARY OF TEST RESULTS

The CAC incinerator is used to treat liquid wastes generated from the chloroacetyl chloride (CAC) and Alachlor processes during the manufacture of agricultural herbicides. It is located at the southeast corner of the Monsanto Chemical Company's John F. Queeny plant in the city of St. Louis, Missouri.

The incinerator system consists of a forced draft, liquid injection burner/thermal oxidizer chamber, quench pot, and an air pollution control scrubber. Liquid CAC Residue is fed by centrifugal pump to the gas-fired incinerator at a maximum rate of 1220 pounds per hour. The heat capacity of the CAC incinerator is 17 million Btu per hour (mmBtu/hr).

The CAC Residue is a liquid waste with a typical density of 1.28 g/mL, viscosity of 4.4 cps, and heat content of 5,700 Btu/lb. It is listed as a corrosive (D002) and reactive (D003) waste. The waste has no water, negligible ash and metals, and is 40-44% chlorides. The primary constituents are acetyl chloride, dichloroacetic anhydride, high boiling tars, chloroacetyl chloride, and several other chlorinated hydrocarbons.

1.1 Process Operation

During the CAC Incinerator trial burn, held from January 31 to February 18, 1989, three (3) waste feed rates were tested. The waste feed rates were chosen to represent Normal, Low, and High input conditions. Oxidizer temperature is then allowed to reach equilibrium with minimal auxiliary fuel (natural gas) usage by means of adjustments in waste feed rate and combustion air flow.

Tables 1-1 through 1-3 present the operating conditions of the incineration process for Test Conditions 1 through 3, which represent sequentially Normal=1, Low=2, and High=3. All process operation measurements shown have been averaged for each test run number; data on variability are shown in later sections of the report. Overall, the process operated smoothly, with coefficients of variation (std dev/mean X 100%) well below 10% in nearly all cases. Within a test condition, the process was operated closely to the first test's established conditions. Nearly all parameters remained within 10% of value from run to run.

Test Condition Normal consisted of a CAC Residue feed rate of 950-1000 lb/hr, oxidizer temperature of 1000-1050°C, and combustion air at 1600-1700 acfm. Test Condition Low consisted of a CAC Residue feed rate of 750-900 lb/hr, oxidizer temperature of 900-1000°C, and combustion air at 1500-1600 acfm. Test Condition High consisted of a CAC Residue feed rate of 1175-1200 lb/hr, oxidizer temperature of 1050-1100°C, and combustion air at 1700-1800 acfm.

TABLE 1-1. SUMMARY OF RESULTS -- PROCESS OPERATION^a -TEST CONDITION NO. 1 - NORMAL

	Run No.					
Parameter	Units	1-1	1-2	1-3		
Test date		1/31/89	2/1/89	2/2/89		
CAC waste feed rate	lb/hr	947.4	994.1	984.8		
Auxiliary fuel feed rate	scfh	790.9 ^b	790.9 ^b	790.9 ^b		
Heat input rate	mmBtu/hr	6.306	6.598	6.509		
Combustion air	acfm	1601	1631	1671		
flow rate Oxidizer temper-	ft/min OC	2039	2077	2129		
ature ^C	-	1025	1049	1034		
Quench outlet	°c					
temperature Scrubber inlet	°c	76.5	79.9	80.4		
temperature		33.9	37.9	38.9		
Quench water flow	gpm					
rate		48.5	47.9	47.8		
Scrubber water	gpm					
flow rate		205.1	205.1	205.1		
Quench/scrubber inlet	рН	8.75	9.32	9.38		
Quench outlet	рH	0.51	0.62	0.73		
Scrubber outlet	pН	1.59	1.82	1.80		
Stack height	ft	50	50	50		
Stack exit velocity	fps	14.2	14.0	13.9		
Exhaust gas	acfm	3134	3080	3061		
flow rate	dscfm	3137	3136	3160		
Stack temperature	°C	10.6	10.0	9.4		
Stack excess 0 ₂ °	8	10.5	10.0	10.3		

a Average of readings taken during each run.
b Assumed values; no gas pressure gauge installed during runs.
C Approximate quench inlet temperature.
d Orsat analysis.

SUMMARY OF RESULTS -- PROCESS OPERATION $^{\mathbf{a}}$ - TEST CONDITION NO. 2 - LOW TABLE 1-2.

	Run No.					
Parameter	Units	2-1	2-2	2-3		
Test date		2/10/89	2/11/89	2/12/89		
CAC waste feed rate	lb/hr	764.5	778.5	895.3		
Auxiliary fuel feed rate	scfh	727.3	790.9	790.9		
Heat input rate	mmBtu/hr	5.183	5.435	6.065		
Combustion air	acfm	1550	1542	1540		
flow rate Oxidizer temper-	ft/min	1975	1964	1962		
ature ^b '		985	988	992		
Quench outlet	°c					
temperature Scrubber inlet	o _C	75.2	75.6	75.6		
temperature Quench water flow	gpm	26.7	26.2	26.2		
rate	31	49.1	49.3	50.6		
Scrubber water	gpm					
flow rate	31	205.1	205.1	205.1		
Quench/scrubber inlet	рН	9.19	9.18	9.10		
Quench outlet	рH	0.76	0.85	0.88		
Scrubber outlet	Hq	2.31	2.33	2.31		
Stack height	ft	50	50	50		
Stack exit velocity	fps	12.6	12.7	12.7		
Exhaust gas	acfm	2772	2811	2805		
flow rate	dscfm	2906	2947	3000		
Stack temperature	°C	6.7	6.7	6.1		
Stack excess 02 ^C	8	10.8	10.8	11.0		

a Average of readings taken during each run.
b Approximate quench inlet temperature.
c Orsat analysis.

TABLE 1-3. SUMMARY OF RESULTS -- PROCESS OPERATION^a - TEST CONDITION NO. 3 - HIGH

	Run No.					
Parameter	Units	3-1	3-2	3-3		
Test date		2/14/89	2/16/89	2/17/89		
CAC waste feed rate	lb/hr	1189.6	1218.9	1202.3		
Auxiliary fuel feed rate	scfh	772.7	681.8	772.7		
Heat input rate	mmBtu/hr	7.693	7.656	7.419		
Combustion air	acfm	1754	1779	1777		
flow rate Oxidizer temper-	ft/min OC	2235	2266	2264		
ature ^b Quench outlet	o _C	1068	1056	1062		
temperature Scrubber inlet	o _C	81.6	82.9	83.7		
temperature Quench water flow	gpm	39.1	39.9	40.0		
rate Scrubber water	gpm	49.3	51.3	54.0		
flow rate	31	205.1	205.1	205.1		
Quench/scrubber inlet	рн	9.30	9.42	9.44		
Quench outlet	pН	0.76	0.62	0.63		
Scrubber outlet	рH	1.73	1.58	1.59		
Stack height	ft	50	50	50		
Stack exit velocity	fps	14.3	13.9	14.1		
Exhaust gas	acfm	3155	3063	3109		
flow rate	dscfm	3322	3273	3324		
Stack temperature Stack excess O ₂ ^C	°C %	6.7 9.6	6.7 10.0	6.1 9.2		

a Average of readings taken during each run.
b Approximate quench inlet temperature.
c Orsat analysis.

1.2 Emissions Performance

Emission performance results are presented in Tables 1-4 through 1-6 for Test Conditions 1 through 3, respectively. Destruction and Removal Efficiency (DRE) results for hazardous constituents in the liquid waste were excellent in all cases, and were 30 to over 100 times better than the regulatory performance standard. Particulate emissions in all cases were very low, at least 2.7 times better than the regulatory performance standard.

Total particulate emissions from the incinerator ranged from 0.32 to 0.65 lb/hr. Emissions of Products of Incomplete Combustion (PICs) were also very low, as indicated by the non-detection (less than 5 ppm) of Carbon Monoxide in the stack gas. Total PIC emissions, as measured by GC/MS wide-scan of organic volatiles and semivolatiles, ranged from 2.29 to 11.45 grams per hour. Sufficient excess air was provided for combustion, as indicated by stack gas O2 concentrations which consistently ranged from 9.2% to 11.0%. These stack gas O2 concentrations represent 75-125% excess air. The HCl removal efficiency of the quench/scrubber section of the incinerator system did not perform quite up to regulatory performance standards. HCl removal efficiencies were 98.8-98.9% for all Test Conditions, with HCl emissions ranging from 1.75 to 8.43 lb/hr.

Table 1-7 presents a summary of averaged test condition trial burn results for major emissions performance and process operation parameters. Each of these parameters is shown in comparison to the permit target.

Figure 1-1 through 1-3 present a summary of the emission testing time periods for Test Conditions 1 through 3, respectively.

SUMMARY OF RESULTS -- EMISSION PERFORMANCE -- TEST CONDITION NO. 1 -- NORMAL TABLE 1-4.

Parameter	Units	1-1	1-2	1-3	Average Std dev CV ^a
Test date DRE - 1,2-Dichloro		1/31/89	2/1/89	2/2/89	
ethane	8	>99.9999	>99.9999	99.9998	99.9999
DRE - Tetrachloro- ethylene	૪	99.9999	99.9996	99.9996	99.9997
Particulates ^b	gr/dscf	0.0295	0.0209	0.0216	0.0240 0.0039 <u>+</u> 16.1%
HCl emissions	lb/hr	6.68	4.48	2.42	4.53 1.74 <u>+</u> 38.4%
HCl removal effic.	8	98.3	99.0	99.4	98.9 0.40 <u>+</u> 0.4%
Stack gas flow rate Oxygen	acfm dscfm %	3134 3137 10.5	3080 3136 10.0	3061 3160 10.3	3092 3144 10.3
Carbon monoxide ^b	ppm	<5	<5	<5	<5
Total PICs	g/hr	2.92	2.83	3.46	3.07 0.34 ±11.1%

a CV = Coefficient of variation.
b Corrected to 7% O₂.

TABLE 1-5. SUMMARY OF RESULTS -- EMISSION PERFORMANCE --TEST CONDITION NO. 2 -- LOW

	Run No.				
Parameter	Units	2-1	2-2	2-3	Average Std dev CV ^a
Test date DRE - 1,2-Dichloro		2/10/89	2/11/89	2/12/89	
ethane	%	99.9999	>99.9999	>99.9999	>99.9999
DRE - Tetrachloro- ethylene	8	>99.9999	99.9999	99.9999	>99.9999
Particulates ^b	gr/dscf	0.0176	0.0223	0.0226	0.0208 0.0023 <u>+</u> 11.0%
HCl emissions	lb/hr	1.75	5.24	4.39	3.79 1.49 <u>+</u> 39.2%
HCl removal effic.	ક	99.5	98.4	98.8	98.9 0.49 <u>+</u> 0.5%
Stack gas flow rate Oxygen	acfm dscfm %	2772 2906 10.9	2811 2947 10.9	2805 3000 10.9	2796 2951 10.9
Carbon monoxide ^b	ppm	<5	<5	<5	<5
Total PICs	g/hr	2.29	2.96	3.79	3.01 0.75 ±24.9%

a CV = Coefficient of variation. b Corrected to 7% 0₂.

TABLE 1-6. SUMMARY OF RESULTS -- EMISSION PERFORMANCE -- TEST CONDITION NO. 3 -- HIGH

			Run No.		
Parameter	Units	3-1	3-2	3-3	Average Std dev CV ^a
Test date DRE - 1,2-Dichloro		2/14/89	2/16/89	2/17/89	
ethane	ક	>99.9999	99.9999	99.9999	99.9999
DRE - Tetrachloro- ethylene	%	99.9999	99.9999	99.9999	99.9999
Particulates ^b	gr/dscf	0.0285	0.0264	0.0286	0.0278 0.0010 <u>+</u> 3.7%
HCl emissions	lb/hr	5.22	8.43	5.27	6.31 1.50 <u>+</u> 23.8%
HCl removal effic.	%	99.0	98.4	99.0	98.8 0.30 ±0.3%
Stack gas flow rate Oxygen	acfm dscfm %	3155 3322 9.7	3063 3273 9.9	3109 3324 9.4	3109 3306 9.7
Carbon monoxide ^b	ppm	<5	<5	<5	<5
Total PICs	g/hr	11.45	7.83	6.57	8.62 2.53 <u>+</u> 29.4%

a CV = Coefficient of variation.
b Corrected to 7% O₂.

TABLE 1-7. SUMMARY OF AVERAGE TRIAL BURN RESULTS AT TEST CONDITIONS

Test condition	Permit target	Normal 1	Low 2	High 3
Oxidizer temperature (OC)	TBD	1036	988	1062
Combustion air flow rate (acfm)	TBD	1635	1544	1770
Stack exit velocity (fps)	TBD	14.0	12.7	14.1
DRE - 1,2-Dichloro ethane (%) DRE - Tetrachloro-	99.99	99.9999	>99.9999	99.9999
ethylene (%)	99.99	99.9997	>99.9999	99.9999
Particulate concentration (gr/dscf)	0.080	0.0240	0.0208	0.0278
HCl emissions (lb/hr) HCl control effic. (%)	4.0 99.0	4.53 98.9	3.79 98.9	6.31 98.8
CO concentration (ppm)	100	<5	<5	<5
O ₂ concentration (%)	TBD	10.3	10.9	9.7
PIC emissions (g/hr)	TBD	3.07	3.01	8.62
Feed rates: (lb/hr) CAC residue Total inorganic ash Total chloride	TBD TBD TBD	975.4 0.49 408.0	812.8 0.45 336.7	1203.6 0.56 500.7
Heat input (mmBtu/hr)	TBD	6.471	5.561	7.589
Atomization pressure (psig)	TBD	109	113	113
Quench water flow rate (gpm)	TBD	48.1	49.7	51.5
Scrubber water flow rate (gpm)	TBD	205.1	205.1	205.1
Utility water pH (S.U.)	TBD	9.15	9.16	9.39

a TBD = To be determined from trial burn data.

Figure 1-1. Summary of Emission Testing Time Periods, Test Condition 1 - Normal.

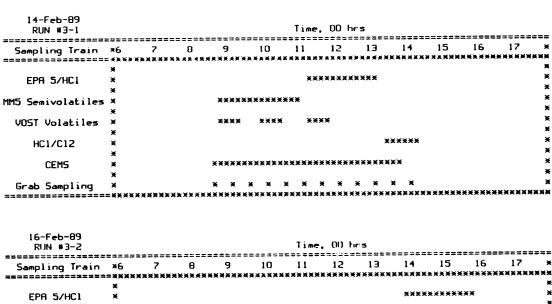
31-Jan-89 RUN #1-1		: Each					, OO I						
Sampling Train	×6	7	8	9	10	11	12	13	14	15	16	17	 *
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EPA 5/HC1	* *				××	*****	****						×
MM5 Semivolatiles	×								×	*****	*****	(XXXXX	×
VOST Volatiles	×				3	***	****	***	*	×××			*
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CAC Waste Off	*	. ~~~~~	~~~~			**	,						×
1-Feb-89 RUN #1-2						Time	., OO t	nrs					
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02.13	* *			****	******	*****	*****	*****	*****	*****	××		×
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Figure 1-2. Summary of Emission Testing Time Periods, Test Condition 2 - Low.

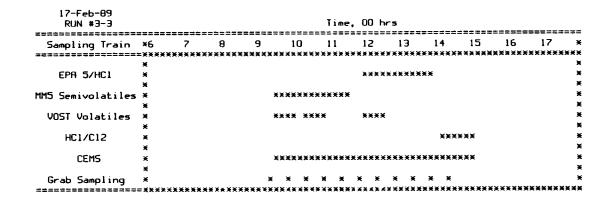
10-Feb-89 RUN #2-1			=====				, oo i	ırs 					
Sampling Train	×6	7	8	9	10	11	12	13	14	15	16	17	×
=======================================	****	(*****	*****	(******	*****	****	*****	(*** *	*****	*****	*****	*****	
EPA 5/HC1	×								¥	*****	****		×
2,11 3,1161	×								_				×
MM5 Semivolatiles	×						*****	*****	××				×
	×												×
VOST Volatiles	×						****	****	*	***			×
HC1/C12	×											****	×
11017012	*											~~~~	×
CEMS	×					***	*****	(******	*****	*****	*****	*****	*
0 1 5 1:	×												*
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		******	****		****	****	****	*****	****	****	****	****	***
11-Feb-89 RUN #2-2							, 00						===
Sampling Train	*6	 7	8	9	10	11	12	13	14	15	16	17	*
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	×												×
EPA 5/HC1	*					**	****	*****					×
MM5 Semivolatiles	×			****	*****	**							× ×
VOST Volatiles	×			XXXX	XXXX	3	EXXX						×
HC1/C12	×								*****				*
CEMS	×			****	******	****	****	*****	*****	•			*
6 1 5 1	*			* *	* * *	* 3	€ ¥ ·	×	. . .				2
Grab Sampling	* -****	*****	*****							-	*****	*****	×××
12-Feb-89 RUN #2-3				=======		Time	-, 00	hrs					
Sampling Train	*6	7	8	9	10	11	12	13	14	15	16	17	•
=============								*****	****	*****	*****	*****	(XX)
	×												3
EPA 5/HC1	×						*****	*****					3
MM5 Semivolatiles	; ×			****	(***** 	€¥¥							3
VOST Volatiles	* *			***	****	×	×××						3
HC1/C12	×							3	****				3
CEMS	×			*****	******	****	*****	****					3

Grab Sampling

Figure 1-3. Summary of Emission Testing Time Periods, Test Condition 3 - High.



RUN #3-2						Tim	e, (O)	tirs	~			======	====
Sampling Train	*6	7	8	9	10	11	12	13	14	15	16	17	*
	 *	****											×
EPA 5/HCl	×								***	****	(**		×
EFH JANCI	×												×
MM5 Semivolatiles	×						****	*****	exx.				×
ing semitorderice	×												×
VOST Volatiles	×			*	***		****	***	XXX:	•			×
	×												*
HC1/C12	×										¥X)	€XXX	×
	×												×
CEMS	×			***	*****	****	****	*****	EXXXXX	****	*****	*** *	*
	×												×
Grab Sampling	×			¥	* *	* *	* ×	* ×	* *	* *	* *	*	*
	=××××	*****	****	*****	*****	****	****	*****	*****	*****	*****	*** **	XXXX



2. INTRODUCTION

The CAC incinerator is located at the Monsanto Chemical Company's John F. Queeny plant in the city of St. Louis, Missouri. The incinerator is used to treat liquid wastes generated from the chloroacetyl chloride (CAC) and Alachlor processes during the manufacture of agricultural herbicides.

The purpose of this report is to submit trial burn results included as part of the Monsanto-Queeny RCRA Part B permit application and to describe how the incinerator system was tested to satisfy the performance requirements specified by the regulations. Test results will be used to establish the operating conditions for the incinerator to assure that performance standards will continue to be met and that human health and the environment will be protected. The trial burn was audited throughout the testing period by Missouri DNR and EPA Region VII representatives.

2.1 Background Information

The incinerator system consists of a forced draft liquid injection burner/thermal oxidizer chamber, quench pot, and an air pollution control scrubber. Liquid wastes were fed by centrifugal pump to the gas-fired incinerator at a maximum rate of 1220 pounds per hour. The incineration system is shown schematically in Figure 2-1 and the system design data summary is provided in Table 2-1. A complete description of the incinerator system and materials of construction was provided in the trial burn plan.

The CAC Residue is a liquid waste with a typical density of 1.28 g/mL, viscosity of 4.4 cps, and heat content of 5,700 Btu/lb. It is listed as a corrosive (D002) and reactive (D003) waste. The waste has no water, negligible ash and metals, and is 40-44% chlorides. The primary constituents are acetyl chloride, dichloroacetic anhydride, high boiling tars, chloroacetyl chloride, and several other chlorinated hydrocarbons. Principal Organic Hazardous Constituents (POHCs) chosen for the trial burn Destruction and Removal Efficiency (DRE) were 1,2-dichloroethane (CAS# 107-06-2) present in the CAC Residue at 0.4-1.5%, and tetrachloroethylene (CAS# 127-18-4) present in the waste at 40-500 ppm. The tetrachloroethylene was spiked up to 2.0% in the CAC Residue for the trial burn test to represent a difficult-to-incinerate hazardous constituent.

Table 2-2 presents the planned test matrix and incineration parameter targets for the trial burn. CAC waste was burned at three (3) waste feed rates representing to normal, low, and high test conditions. Good agreement between the major process parameters -- waste feed rate, temperature, and combustion air -- was maintained according to the trial burn plan. Other process parameters are typically set according to temperatures in the quench/scrubber sections which protect materials of construction.

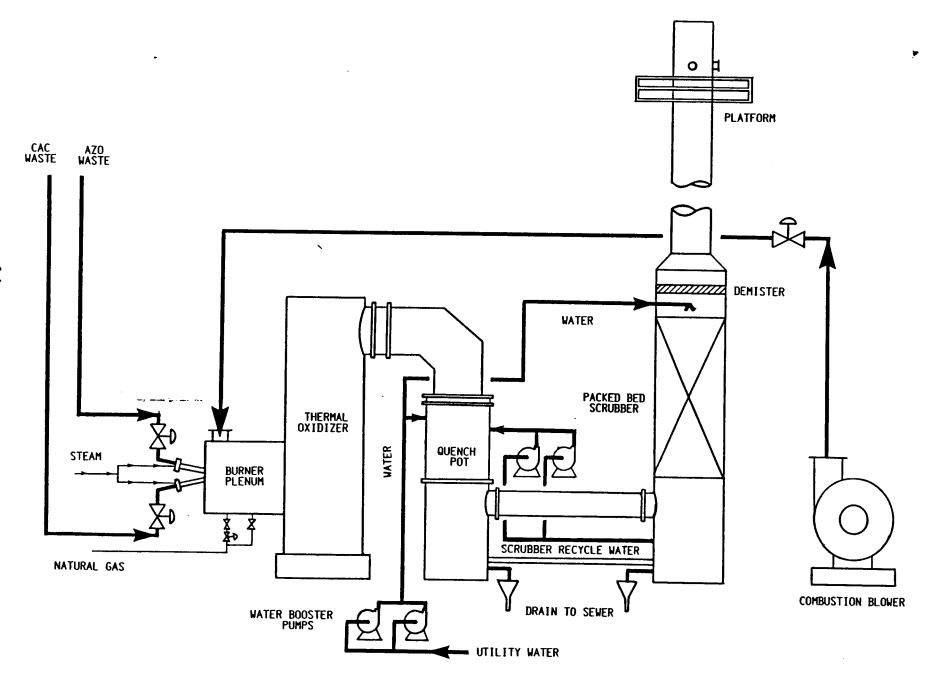


Figure 2-1. Schematic Diagram of the CAC Incinerator System at Monsanto-Queeny Plant.

TABLE 2-1. SUMMARY OF DESIGN INFORMATION FOR MONSANTO-QUEENY CAC INCINERATOR

Parameter	Units	Combined system
Type of incinerator		Liquid fired burners to vertical thermal oxidizer
Inside dimensions (diameter x length)	ft	Burner plenum = 3.0 x 3.0 Oxidizer = 5.792 x 21.906 Breeching duct = 2.875 x 14.617
Cross-sectional area	ft ²	Burner plenum = 7.07 Oxidizer = 26.35 Breeching duct = 6.49
Volume, inner	ft ³	693.3
Heat capacity	10 ⁶ Btu/hr	17.0
Refractory thickness	inches	Burner plenum = 9.0 Oxidizer = 4.5 Breeching duct = 3.0
Refractory conductivity	Btu-in./ hr-ft ² - ⁰ F	unknown (assume 5% heat loss)
Refractory surface area	ft ²	593.8 (all surfaces)
Cooled surface area	ft ²	0
Waste feed system		Liquid injection with steam atomization
Installation date	year	1976
Blower/fan capacity	scfm	4000
Maximum quench inlet temperature	$o_{\mathbf{F}}$	2000
Maximum scrubber inlet temperature	$o_{\mathtt{F}}$	190
HCl removal capacity	lb/hr	540 (1400 lb/hr @ 39% Cl)
Quench water supply maximum capacity	gpm	Utility water = 100 Scrubber recycle = 75
Quench water temperature	o _F	Utility water = 60 Scrubber recycle = 176

TABLE 2-2. SUMMARY OF TEST CONDITION PARAMETER TARGETS FOR CAC INCINERATOR SYSTEM

		Test	condition	
Incinerator sys	stem	1-Normal	2-Low	3-High
[Note: Normal t	ype = targo	et value; Bold	type = actu	al test value]
Waste type(s)		CAC	CAC	CAC
Operation mode		normal	low	maximum
Waste feed rate (lb/hr)	•	1100 975 753-1020	800 ^a 813 680-912	1400 ^a 12 04 1135–1318
POHC feed rate (lb/min)	PCE	0.367 0.369	0.267 0.298	0.467 0.388
	1,2-DCE	0.330 0.059	0.240 0.097	0.420 0.218
Chloride feed r (lb/hr)	ate	407 408	296 337	518 501
Ash feed rate (lb/hr)		TBD 49	TBD 45	TBD 56
Waste heat input (mmBtu/hr)	ıt	9.086 6.471	6.608 5.561	11.564 7.589
Auxiliary fuel (mmBtu/hr)		2.275 0.870	2.275 0.847	2.275 0.817
Exhaust gas flo	w rate	3300 3144	3300 2951	3300 3306
Oxidizer temper	ature	980 - 1040 1036	900 - 950 988	1050-1080 1062
O ₂ in stack gas (%)	;	TBD ^b 10.3	TBD 10.9	TBD 9.7
CO in stack gas (ppmv)	1	max 500 <5	max 500 <5	max 500 <5

 $^{^{\}rm a}$ Will become set according to oxidizer temperature. $^{\rm b}$ To be determined during trial burn tests.

TABLE 2-2. SUMMARY OF TEST CONDITION PARAMETER TARGETS FOR CAC INCINERATOR SYSTEM (Continued)

	5	Cest o	condition	
Incinerator system parameter	1-Nor	nal	2-Low	3-High
[Note: Normal type = target	t value;	Bold	type = actual	test value]
Quench water flow (gpm)	25 48		25 50	25 52
Scrubber water flow (gpm)	140 205		140 205	140 205
Scrubber recycle flow (gpm)	50 43		50 45	50 46

a Will become set according to oxidizer temperature.

b To be determined during trial burn tests.

2.2 Non-Standard Events/Conditions

Most large test programs have changes, unanticipated events, and improvements occur as the program tasks proceed according to the test plan document. Changes in this trial burn test program included a change in type and model of waste flowmeter, recalibration of the waste flowmeter based on new information, one mid-test waste shutoff, addition of extra test runs, addition of a new sampling/analysis method for speciation of HCl and $\rm Cl_2$ emissions, extra waste feed chloride analyses, addition of metals analyses for the waste, and an improved waste analysis method for water content.

During Test Condition 1, a Signet Model 8500 open-cell paddlewheel flow sensor/transmitter was used to measure CAC Residue feed rate to the incinerator. The Signet unit had been calibrated according to the calibration table supplied with the unit and a liquid waste specific gravity of 1.12. At the beginning of Test Condition 2 before process and emission testing started, the Signet flow meter malfunctioned and testing was postponed until correction. After one week, a new flow meter was installed and calibrated, which was an improvement to a Rosemont

Model 8711 Magnetic Flowtube and Model 8712 Transmitter. In discussions with Signet technical representatives, it was learned by the Queeny instument engineers that the wrong calibration table had been supplied with the unit. A correct calibration table which relates the constants for pulses/gallon and pipe I.D. was received, and the actual waste feed rates for Test Condition 1 were back-calculated. Test Conditions 2 and 3 as well as extra HCl/Cl₂ emission test runs were then conducted with the Rosemont pulsed DC magnetic flow meter operating.

During Test Condition 1 - Run 1, the waste feed was inadvertently shutoff by the process operator to conduct the waste feed auto shutoff check required under 40 CFR 264.347(c). The shutoff and return lasted approximately 25-35 minutes before full waste feed rate was achieved. An additional VOST sampling run was performed to compensate for the interrupted volatile emissions test. The particulate/chloride test run (#5-1-1) continued throughout the interruption.

An extra sampling test method was added to the program to collect separately HCl and ${\rm Cl}_2$ emissions for analysis. The standardized EPA Method 5 sampling train with chloride absorption in alkaline liquid media in the impinger back-half portion measures only total chlorides in the stack gas, because the absorbing solution converts both HCl and any free chlorine (${\rm Cl}_2$) to chloride ions for subsequent analysis. A draft EPA test method was used which separately traps HCl in an acidic solution while allowing any free ${\rm Cl}_2$ to pass through for absorption and conversion to chloride in an alkaline solution. This method was added because of the potential for ${\rm Cl}_2$ formation in the incinerator when burning a highly chlorinated waste stream. Analysis of both fractions is performed using ion chromatography.

Other analytical changes included the addition of analyzing each test run's CAC Residue for chloride content, adding six metals (Sb, Ba, Be, Hg, Ag, Tl) to the once per test run waste analyses to allow for screening against future metals emissions regulations for hazardous waste incinerators, and a change to Karl-Fischer analysis for water content in the CAC Residue due to water reactivity with the analytical QC spike required by the ASTM D95 xylene codistillation method.

3. PERFORMANCE RESULTS

RCRA regulatory performance standards for hazardous waste incinerators [40 CFR 264.343] require that:

- Destruction and Removal Efficiency be greater than 99.99% for selected most-difficult-to-incinerate Principal Organic Hazardous Constituents in the waste stream [264.343(a)];
- 2. Particulate emissions be less than 0.08 grains (7000 grains per pound) per dry standard cubic foot (gr/dscf) of exhaust gas when corrected to a standardized value of 7% oxygen in the stack gas [264.343(b)]; and
- 3. HCl emissions be less than the larger of either 4 lb/hr of 1% of the HCl in the stack gas (99% control efficiency) prior to entering any air pollution control equipment [264.343(c)].

3.1 POHCs/DRE

Principal Organic Hazardous Constituents (POHCs) chosen for the trial burn Destruction and Removal Efficiency (DRE) were 1,2-dichloroethane (CAS# 107-06-2) present in the CAC Residue at 0.4-1.5%, and tetrachloroethylene (CAS# 127-18-4), also present in the waste at 40-500 ppm. The tetrachloroethylene (also called perchloroethylene, PCE, and tetrachloroethene) was spiked up to 2.0% by weight in the CAC Residue for the trial burn test to represent a difficult-to-incinerate hazardous constituent.

Table 3-1 presents a summary of the measured DREs for the chosen POHCs in this trial burn. Each test run number represents the average of two to four VOST emission tests per test run. DRE results for 1,2-dichloroethane ranged from 99.9998% to >99.9999%, which corresponds to at least 54 times the regulatory performance standard [264.343(a)]. In one-third of the tests, 1,2-DCE was not detected in the stack gas (<0.23 ug/m³ or 0.06 ppb). Perchloroethylene DRE results ranged from 99.9996% to >99.9999%, which corresponds to at least 30 times the regulatory performance standard. At MDNR's request, DRE of PCE was attempted using the semivolatile MM5 sampling train; no valid DRE results were obtained.

Table 3-2 presents the CAC Residue POHC analysis results with calculation of weight percent of each POHC for each test run's waste sample. The trial burn plan for spiking the waste to a target concentration of 2% PCE was maintained, as shown in this table. Concentrations of POHCs held steady within each test condition, as indicated by the low coeffficients of variation (CV) for each test condition.

TABLE 3-1. SUMMARY OF POHC DESTRUCTION AND REMOVAL EFFICIENCY (DRE), CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond	Run No.	POHC Fee (Win), o 1,2-DCE	g/min	POHC Em: (Wout), 1,2-DCE	mg/min	DRE 1,2-DCE	, % PCE
							wa-
1	1-1	23.62	143.89	<0.0134	0.0167	>99.9999	99.9999
Normal	1-2		180.34	<0.0129	0.7960	>99.9999	99.9996
	1-3		177.22	0.0456	0.5947	99.9998	99.9996
	Avg	26.58	167.15				-th -th
	CV	13.3%	9.9%				
2	2-1	39.90	128.38	0.0490	<0.0184	99.9999	>99.9999
Low	2-2	37.69	121.31	<0.0191	0.1328	>99.9999	99.9999
	2-3	54.80	156.29	<0.0201	0.0215	>99.9999	99.9999
	Avg	44.13	135.33				
	CV	17.2%	11.2%				
3	3-1	102.55	152.03	<0.0216	0.0376	>99.9999	99.9999
High	3-2	98.60	186.14	0.0819	0.0877	99.9999	99.9999
-	3-3	95.41	189.91	0.0753	0.0995	99.9999	99.9999
	Avg	98.85	176.03	0.0596	0.0749		
	CV	3.0%	9.7%	45.3%	35.8%		

TABLE 3-2. CAC RESIDUE POHC ANALYSIS RESULTS, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond	Run No.	Concentration 1,2-DCEa	on, mg/L PCE	Density g/mL	Concentrati 1,2-DCE	on, wt% ^C PCE
1	W-1-1 W-1-2	4,300 5,400	26,000 31,000	1.291 1.290	0.33 0.42	2.01
	W-1-3	4,270	30,700	1.289	0.33	2.38
	Avg	4,657	29,233	1.290	0.36	2.26
	CV	11.3%	7.8%	0.1%	11.8%	7.9%
2	W-2-1	8,800	28,500	1.283	0.69	2.22
	W-2-2	8,200	26,300	1.279	0.64	2.06
	W-2-3	10,300	29,500	1.277	0.81	2.31
	Avg	9,100	28,100	1.280	0.71	2.20
	CV	9.7%	4.8%	0.2%	10.0%	4.7%
3	W-3-1	14,600	21,700	1.283	1.14	1.69
	W-3-2	13,700	25,900	1.282	1.07	2.02
	W-3-3	13,500	26,800	1.285	1.05	2.09
	Avg	13,933	24,800	1.283	1.09	1.93
	CV	3.4%	9.0%	0.1%	3.6%	9.0%

a_{1,2-DCE} = 1,2-Dichloroethane
b_{PCE} = Tetrachloroethene
C_{wt} = mg/L X E-3g/mg X E-3L/mL X g/mL X 100%

Table 3-3 presents the calculations of POHC feed rate (W_{in}) needed for compute DRE for each test run. Again, maintenance of steady feed rate of each POHC within a test condition was excellent, as indicated by the low coefficients of variation (3 to 17%). Table 3-4 presents the calculation of POHC emission rate for each test run, based on the 2-4 VOST samples collected during each test run. Variability in emission rate of a POHC is larger due to the occasional non-detection (<5 ng per sample) in the stack gas. For averaging purposes, the emission rate at the level of detection was used in all applicable calculations.

TABLE 3-3. CALCULATION OF POHC FEED RATE $(w_{i\,n})$, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond	Run No.	CAC Waste Feed Rate lb/hr	POHC Cont 1,2-DCE	ent, wt% PCE	POHC Feed Rat 1,2-DCE	ce, lb/hr PCE
1 Norma	1-1 1 1-2 1-3	947 994 985	0.33 0.42 0.33	2.01 2.40 2.38	3.13 4.17 3.25	19.03 23.86 23.44
	Avg CV	975 2.1%	0.36 11.8%	2.26 7.9%	3.52 13.3%	22.11
2 Low	2-1 2-2 2-3	765 779 895	0.69 0.64 0.81	2.22 2.06 2.31	5.28 4.99 7.25	16.98 16.05 20.67
	Avg CV	813 7.2%	0.71 10.0%	2.20 4.7%	5.84 17.2%	17.90 11.2%
3 High	3-1 3-2 3-3	1190 1219 1202	1.14 1.07 1.05	1.69 2.02 2.09	13.57 13.04 12.62	20.11 24.62 25.12
	Avg CV	1204	1.09	1.93 9.0%	13.08 3.0%	23.29 9.7%

TABLE 3-4. CALCULATION OF POHC EMISSION RATE (W_{out}), CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test	Run	POHC Stac		Stack Gas Flow Rate	POHC E	mission
Cond	No.	1,2-DCE	PCE	dscfm	1,2-DCE	
1	1-1A	<0.150	0.300	3,137	<0.0133	0.0266
Normal		<0.148	<0.148	3,137	<0.0131	<0.0131
	1-1C	<0.150	<0.150	3,137	<0.0133	<0.0133
	1-1D	<0.154	<0.154	3,137	<0.0137	<0.0137
	1-1 Avg	<0.151	0.188		<0.0134	0.0167
	CV	1.4%	34.4%		1.4%	34.4%
	1-2A	<0.146	16.588	3,136	<0.0130	1.4730
	1-2B	*	*	3,136	NA	NA
	1-2C	<0.144	1.340	3,136	<0.0128	0.1190
	1-2 Avg	<0.145	8.964		<0.0129	0.7960
	CV	0.7%	85.1%		0.7%	85.18
	1-3A	<0.142	0.331	3,160	<0.0127	0.0296
	1-3B	0.878	12.961	3,160	0.0786	1.1598
	1-3C	*	*	3,160	NA	NA
	1-3 Avg	0.510	6.646	-	0.0456	0.5947
	CV	72.2%	95.0%		72.2%	95.09
2	2-1A	1.339	<0.223	2,906	0.1102	<0.0184
Low	2-1B	<0.223	<0.223	2,906	<0.0184	<0.0184
	2-1C	<0.223	<0.224	2,906	<0.0184	<0.0184
	2-1 Avg	0.595	<0.223		0.0490	<0.0184
	CV	88.4%	0.2%		88.4%	0.28
	2-2A	<0.232	0.418	2,947	<0.0194	0.0349
	2-2B	<0.235	4.184	2,947	<0.0194	0.3448
	2-2C	<0.223	<0.223	2,947	<0.0186	<0.0186
	2-2 Avg	<0.229	1.591		<0.0191	0.1328
	CV	1.9%	113.0%		1.9%	113.08
	2-3A	<0.237	0.285	2,955	<0.0198	0.0238
	2-3B	<0.239	<0.239	2,955	<0.0200	<0.0200
	2-3C	<0.248	<0.248	2,955	<0.0208	<0.0208
	2-3 Avg	<0.241	0.257		<0.0202	0.0215
	CV	2.2%	8.0%		2.2%	8.09

^{*}Sample broken during shipment to laboratory.

TABLE 3-4. CALCULATION OF POHC EMISSION RATE (W_{out}), CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT (Continued)

		POHC Stac	k Gas	Stack Gas	POHC E	mission
Test	Run	Concentration		Flow Rate		
Cond	No.	1,2-DCE	PCE	dscfm	1,2-DCE	
3	3-1A	<0.231	0.694	3,322	<0.0217	0.0653
High	3-1B	<0.237	0.285	3,322	<0.0217	0.0267
5	3-1C	<0.227	<0.227	3,322	<0.0214	<0.0214
	0 10					
	3-1 Avg	<0.232	0.402		<0.0216	0.0378
	cv	0.8%	52.3%		0.8%	52.3%
	3-2A	1.064	2.683	3,273	0.0986	0.2487
	3-2B	2.004	0.638	3,273	0.1857	0.0591
	3-2C	<0.238	<0.238	3,273	<0.0221	<0.0221
	3-2D	<0.228	0.228	3,273	<0.0211	0.0211
				·····	 	
	3-2 Avg	0.884	0.947		0.0819	0.0877
	cv	82.7%	107.3%		82.7%	107.3%
	3-3A	0.908	2.496	3,324	0.0855	0.2349
	3-3B	1.310	0.468	3,324	0.1233	0.0440
	3-3C	<0.222	<0.222	3,324	<0.0209	<0.0209
				<u> </u>	· <u></u>	
	3-3 Avg	0.813	1.062		0.0766	0.0999
	cv	54.3%	96.6%		54.3%	96.6%

^{*}Sample broken during shipment to laboratory.

3.2 <u>Chlorides</u>

Emissions of chlorides are dependent on the input of chlorinated molecules fed to the incinerator. The CAC Residue averaged 41.65% chloride with a CV of $\pm 2.9\%$. Table 3-5 presents a summary of the CAC Residue characterization data for each test run.

HCl emission control in the CAC Incinerator is provided both by the water quench which cools the stack gas and by water absorption in the packed column absorber/scrubber. A portion (about 20%) of the scrubber discharge is recycled to the quench section to make 45% of the water entering the quench throat.

Two different measurement techniques were used to determine chloride emissions from the CAC Incinerator. Total chlorides in the stack gas were measured using the EPA Method 5 sampling system with back-half collection of chlorides by alkaline absorption and analysis by ion chromatography for the chloride ion. Hydrogen chloride (HCl) and chlorine gas (Cl₂) in the stack gas were measured using a draft EPA test protocol for HCl emissions. The principle of the method is that it differentiates between HCl and Cl₂ by first preferentially absorbing HCl in acidic solution while Cl₂ passes through to be then absorbed and converted to chloride ion in an alkaline solution. Both halves are then analyzed by ion chromatography (EPA Method 300.0) for chloride ion concentration.

Table 3-6 presents a summary of the total chloride and $\mathrm{HCl/Cl_2}$ emission results. It shows that the total chloride emissions (as measured by EPA 5/Cl) from the incinerator remained steady with a low CV for each test condition. An equal amount of HCl and $\mathrm{Cl_2}$ is emitted during the Low and Normal test conditions, with a shift to a 60:40 $\mathrm{Cl_2}$:HCl ratio under the High waste feed rate condition. A distinction in total chlorides and HCl is made in Table 3-6. HCl emissions are calculated by multiplying the chloride results by the formula weight ratio $[\mathrm{HCl/Cl:}(36.461/35.453)]$.

Table 3-7 presents the summary of HCl scrubber removal efficiency for each test run. HCl emission control averaged 98.8 to 98.9% throughout the trial burn.

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TABLE 3-5. SUMMARY OF CAC RESIDUE CHARACTERIZATION DATA, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

						Di N.					
Parameter	Units	1-1	1-2	1-3	2-1	Run No. 2-2	2-3	3-1	3-2	3-3	Avg C
Heat Value	 8tu/lb	5,740	5,763	5,725	5,730	5,860	5,805	5,750	5,665	5,465	5,723 1.
Water Content	wt %	<0.02	<Ó.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Ash	wt %	0.05	0.05	0.05	0.07	0.06	0.04	0.06	0.03	0.05	0.05 21.
Viscosity @ 19 C	cps	4.4	4.3	4.4	4.4	4.4	4.4	4.3	4.4	4.4	4.4 0.
Density @ 19 C	g/mL	1.291	1.290	1.289	1.283	1.279	1.277	1.283	1.282	1.285	1.284 0.
Total Chlorides	ώt %	41.44	42.06	41.96	44.40	40.15	40.00	42.19	41.26	41.37	41.65 2
Carbon	wt %	NA	33.25	NA	NA	33.18	NA	NA	30.58	NA	32.34 3.
Hydrogen	wt %	NA	2.50	NA	NA	4.04	NA	NA	3.17	NA	3.24 19.
0xygen	wt %	NA	22.05	NA	NA	22.57	NA	NA	24.98	NA	23.20 5.
Nitrogen	wt %	NA	<0.01	NA	NA	<0.01	NA	NA	<0.01	NA	<0.01
Sulfur	wt %	NA	<0.01	NA	NA	<0.01	NA	NA	<0.01	NA	<0.01
Total Organic Halogen	g/L	471	358	122	123	180	132	141	45.3	31.7	178 76.
(by EPÄ Method 9020)	wt %	36.48	27.75	9.46	9.59	14.07	10.34	10.99	3.53	2.47	13.85 76.
Antimony (Sb)	mg/L	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55
Arsenic (As)	mg/L	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030	<0.030
Barium (Ba)	mg/L	<0.046	<0.046	<0.046	0.060	0.054	0.072	0.076	<0.046	0.054	0.056
Beryllium (Be)	mg∕L	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018
Cadmium (Cd)	mg/L	<0.054	<0.054	<0.054	<0.054	<0.054	<0.054	<0.054	<0.054	<0.054	<0.054
Chromium (Cr)	mg/L	0.53	0.60	0.65	10.5	0.69	0.74	8.35	3.41	1.39	2.98 13
Lead (Pb)	mg/L	0.074	0.066	0.22	0.61	0.074	0.60	0.38	<0.018	<0.018	0.23
Mercury (Hg)	mg/L	<0.0012	<0.0014	0.0022	< 0.0012	<0.0012	<0.0012	<0.0012	< 0.0012	<0.0012	0.0013
Silver (Ag)	mg/L	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08
Thallium (T1)	mg/L	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00

NA = Not analyzed

TABLE 3-6. SUMMARY OF TOTAL CHLORIDE AND HC1/C1₂ EMISSION RESULTS

		ED35/01 Emain	1101 /0	llo Mania	Mo±o1	Chloridos	_
Test	Run	EPA5/Cl Train HCl	HC1/C	Cl ₂		Chlorides HCl/Cl ₂	
				lb/hr	lb/hr	lb/hr	∂111 %D
Cond	No.	lb/hr	lb/hr	ID/III	15/111	15/111	<u>ل</u> وه
1	1-1	8.77	6.68	5.53	8.53	12.02	33.9%
Normal	1-2	9.84	4.48	5.03	9.57	9.38	1.9%
	1-3	9.67	2.42	4.37	9.41	6.72	33.3%
	Avg	9.43	4.53	4.98	9.17	9.37	2.2%
	CV	5.0%	38.4%	9.5%	5.0%	23.1%	
2	2-1	10.38	1.75	2.12	10.10	3.82	90.1%
Low	2-2	10.35	5.24	4.06	10.06	9.15	9.5%
	2-3	10.28	4.39	5.22	10.00	9.48	5.3%
	Avg	10.34	3.79	3.80	10.05	7.49	29.3%
	CV	0.4%	39.2%	33.7%	0.4%	34.6%	
3	3-1	12.72	5.22	7.83	12.37	12.91	4.2%
High	3-2	13.76	8.43	10.29	13.38	18.49	32.0%
J	3-3	13.42	5.27	9.34	13.05	14.47	10.3%
	Avg	13.30	6.31	9.15	12.93	15.29	16.7%
	CV	3.3%	23.8%	11.1%	3.3%	15.4%	

TABLE 3-7. SUMMARY OF HC1 EMISSION CONTROL EFFICIENCY, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond	Run No.	Total Chloride Feed Rate lb/hr	Total HCl Emiss Rate lb/hr	HCl Emission Rate lb/hr	HCl Removal EPA5/Cl	Efficiency HC1/C1 ₂
1	1-1 1-2	392.44	8.77	6.68	97.8%	98.3% 99.0%
Normal	1-3	418.08 413.31	9.84 9.67	4.48 2.42	97.7% 97.7%	99.4%
	Avg CV	407.94	9.43 5.0%	4.53 38.4%	97.8% 0.1%	98.9%
2 Low	2-1 2-2 2-3	339.66 312.77 358.00	10.38 10.35 10.28	1.75 5.24 4.39	97.0% 96.8% 97.2%	99.5% 98.4% 98.8%
	Avg CV	336.81 5.5%	10.34	3.79 39.2%	97.0% 0.2%	98.9%
3 High	3-1 3-2 3-3	502.06 502.96 497.27	12.72 13.76 13.42	5.22 8.43 5.27	97.5% 97.3% 97.4%	99.0% 98.4% 99.0%
	Avg CV	500.76 0.5%	13.30 3.3%	6.31 23.8%	97.4%	98.8%

Notes: Total chlorides measured by EPA5/Cl sampling train and total Cl analysis by IC.

"HCl only" measured by ${\rm Cl}^-/{\rm Cl}_2$ sampling train with separate chloride analyses by IC.

3.3 Particulates

Table 3-8 presents the particulate emission test results, both on an "as measured" basis and corrected to 7% O_2 in the stack gas [gr/dscf @ 7% O_2 = gr/dscf X $(14/21-O_2\%)$]. The CAC Incinerator performed at least 2.7 times better than the regulatory performance standard for particulate emissions while burning CAC Residue. The highest particulate emissions occurred during the inadvertent waste feed shutoff of Test Run #5-1-1, under the Normal waste feed rate condition. Particulate emissions during the High waste feed rate condition averaged the highest, but were never as high as Test Run #5-1-1. The ash concentrations in the CAC Residue were very small, ranging from 0.03 to 0.07%.

TABLE 3-8. SUMMARY OF PARTICULATE EMISSION TEST RESULTS, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond.	Run No.	Oxygen Conc avg %	Particulate As Measured gr/dscf	Concentration At 7% O ₂ gr/dscf	Emission Rate lb/hr
1 Normal	5-1-1 5-1-2 5-1-3	10.5 9.9 10.0	0.0221 0.0166 0.0170	0.0295 0.0209 0.0216	0.609 0.445 0.448
	Avg CV	10.1	0.0186 13.5%	0.0240 16.1%	0.501 18.7%
2 Low	5-2-1 5-2-2 5-2-3	10.9 10.9 10.7	0.0127 0.0161 0.0166	0.0176 0.0223 0.0226	0.317 0.399 0.413
	Avg CV	10.8	0.0151 11.4%	0.0208 11.0%	0.376 13.7%
3 High	5-3-1 5-3-2 5-3-3	9.7 9.8 9.6	0.0230 0.0211 0.0233	0.0285 0.0264 0.0286	0.645 0.591 0.650
	Avg CV	9.7 0.8%	0.0225 4.3%	0.0278 3.7%	0.629 5.2%

Note: %02 as measured by Orsat analysis of concurrent integrated bag gas samples.

4. PROCESS OPERATING CONDITIONS

The principal components of the Monsanto Chemical Company's Queeny plant CAC incinerator were designed and fabricated by the John Zink Company of Tulsa, Oklahoma. Built circa 1976 under Service Order #081181, it is a design with no model number.

4.1 Process Overview

The incinerator is designed as a combination liquid injection and gas thermal oxidizer, consisting of a horizontal burner plenum, vertical oxidizer chamber, water quench pot, and water absorber (scrubber). Waste gases are no longer burned in the unit.

Auxiliary fuel (natural gas) is used to bring the oxidizer up to minimum operating temperature (about 850°C) before the waste streams are introduced and maintain the correct operating temperature (980°C) under normal operating conditions. The liquid waste enters the system by way of special patented burners under pressure with auxiliary steam to assure complete atomization. When fired, a high temperature oxidizing region is formed through which the waste must pass. The waste is thermally dissociated and then oxidized with an excess of combustion air. The furnace is sized to insure sufficient residence time for all reactions to go to completion. A minimum of auxiliary fuel is used to maintain stable burning temperatures for the waste streams.

The oxidizer unit is a vertically oriented, self-supported unit. Gaseous reaction products and inerts exit near the top of the oxidizer and are directed downward through a specially designed aqueous quench system. The gas stream is quenched from design temperatures of 980°C to 88°C, then directed to a combination absorption column and vent stack. Hydrogen chloride is removed from the combustion gas stream by means of a countercurrent aqueous stream in a packed absorption column.

A schematic diagram of the CAC incinerator was shown in Figure 2-1. The incinerator is a "forced draft" type unit, in that the prime mover is the combustion air blower. Key incinerator design information was summarized in Table 2-1.

The incinerator section of the system consists of a burner plenum followed by a thermal oxidizer chamber. The burner plenum has outside dimensions of 4.33 ft. in diameter by 6.0 ft. in vertical length. Inside the plenum, combustion air enters through a 1.208 ft. diameter tangential duct, where it meets 20 spin vanes. Waste burner guns enter horizontally at angles through the end of the plenum and extend to the refractory tile section of the plenum. The plenum refractory section consists of an 11 inch I.D. entry to a cylindrical chamber which is 3.0 ft. long by 3.0 ft. I.D., and exit through a 9 inch long by 2.042

inch I.D. connecting duct. The refractory tiles are C.E. Chemal 85B fire brick or equivalent.

The natural gas pilot assembly protrudes into the front of the tiled plenum along with scanner/control nozzles and an auxiliary gas nozzle.

The thermal oxidizer section stands vertically and is 23.344 ft. high by 7.0 ft. in diameter outside dimensions. The inlet duct is centered 3.344 ft. above the chamber outside floor. Inside dimensions are 22.014 ft. high by 5.792 ft. in diameter for the refractory lined oxidizer chamber. Gases leave the oxidizer through a 2.875 ft. I.D. refractory lined breeching duct near the top of the chamber. Other connections and nozzles to the thermal oxidizer chamber include water/steam tempering guns, sight port, waste gas feed nozzle (not used), and manway. Refractory limits are rated at 2900°F (1600°C).

The waste feed to the incinerator goes through the John Zink Model HI-24 burner assembly. Both CAC and nonhazardous Azo residues are fed to the burner plenum using a "DH" waste gun insert. CAC residue is normally fed at 1000 lb/hr (about 1.5-1.6 gpm) liquid at 65 psig at the gun tip, with atomizing steam at 110 psig. The CAC waste recirculation line carries about 60 gpm back to the CAC residue storage tank. When fired, Azo residue is fed at 275 lb/hr liquid at 50 psig at the gun tip, with atomizing steam at 80 psig. Provision is made in the HI-24 assembly for two other burner guns, one for auxiliary fuel oil (not used) and another for a former liquid waste stream (not used).

The CAC Residue flow rate is measured using a Rosemont Model 8711 Flowtube and Model 8712 Transmitter, installed in a 0.5-inch I.D. teflon-lined pipe run. The magnetic flow meter operates on the "pulsed DC" principle for fluids with ≥ 5 micromhos/cm flow conductivity, and has a flow rate range of 0.02 to 30 ft/sec. Ambient temperature operating limits are from -20° to $+130^{\circ}$ F, with an output signal from 4 to 20 mA, internally powered, on a 0-1000 ohm load. Accuracy is ± 0.5 % of rate from 1-30 ft/sec; between 0.02-1.0 ft/sec, the system has an accuracy of ± 0.005 ft/sec. Repeatability is ± 0.1 % of reading, with a response time of 0.4 sec maximum response to a step change in input. The flow meter ha a stability of ± 0.1 % of rate over six months, has an ambient temperature effect of ± 1 % of reading per $\pm 100^{\circ}$ F, and has a radio frequency interference effect of ± 0.05 % of span at 3 V/m.

The auxiliary fuel gas enters the burner plenum through the side, adjacent to the gas pilot assembly. The pilot is rated at 380,000 Btu/hr natural gas, and the auxiliary gas burner is rated at 5,500,000 Btu/hr. Fuel gas specifications include 910 Btu/scf, 0.55 specific gravity, 30 psig available, and 10 psig at the burner.

The combustion air blower is a Garden City Blower Model No. 325-6-40 powered by a 40 hp electric motor operated at 250 rpm to

push the combustion gases through the incinerator system. Maximum design combustion air requirements are 3300 scfm at 30 in. $\rm H_2O$ static pressure and $100^{\rm O}\rm F$ ambient with 25% excess air combustion requirement. This blower has a nominal capacity of 4000 scfm at $100^{\rm O}\rm F$. The combustion air flow can be controlled by a damper valve, which is instrumented to respond automatically or manually. A low flow switch assures adequate air for safe operation.

From near the top of the oxidizer, the exhaust gases are directed out and down to the quench pot through a 3.583 ft. O.D. refractory-lined breeching duct (2.875 ft. I.D.). The quench pot supports a contact tube, water weir and aqueous quench gun assembly. Approximately 100 gpm of water are used to quench the exhaust gases through eight quench water guns located on the quench contactor circumference, four each at two levels. The upper ring of quench guns uses water supplied from the boosted utility water header (about 50 gpm), and the lower quench ring uses water supplied from the scrubber recycle pumps (about 50 gpm), as shown in Figure 2-1. The quench contactor section is 4.0 ft. high by 4.0 ft. I.D., and is refractory-lined to a final inner diameter of 3.25 ft.

The quench pot is 4.0 ft. I.D. by 9.604 ft. high, with a downcomer that extends 2.937 ft. into the pot section. Cooled exhaust gases leave the quench pot near the top past the downcomer through a 2.167 ft. I.D. duct. Quench water is discharged from the quench pot through a drain connection to the plant sewer.

2.167 ft. I.D. by 16.833 ft. connecting duct routes exhaust gases from the quench pot to the packed tower absorber. The lower absorber section is 5.0 ft. I.D. by 26.5 ft. high. From the 10.5 ft. high mark to the 20.0 ft. mark (9.5 ft.) is the packed tower absorber section, consisting of a Norton #22808 ceramic packing support plate, 59 inches O.D., and 200 ft³ of 2inch tel-zell Glitsch saddles (1/8-inch wall thickness). 150-200 gpm of water flows through a 3-inch teflon-lined exterior nozzle pipe directed down onto a Norton titanium distributor above the packed tower section. Approximately 50 gpm scrubber effluent water is recycled to the quench throat. The remaining scrubber water is discharged from the lower absorber section through a drain connection to the plant sewer. The absorber and quench pot are connected by a 4-inch line for liquid level equalization.

A 2.167 ft. I.D. stack is located above the scrubber/absorber, necked down from 5.0 ft. I.D. at the 28 ft. high mark. Stack exit is 50.0 ft. above ground level. Sampling and gas monitoring ports are located at 45.0 ft. above ground level with a service platform surrounding 180° of the circumference and located at 40.0 ft. above ground level.

The process control sensors and monitors described in Table 4-1 are used at the facility. The location of the sensors is

shown in Figure 4-1; more detailed location information can be found by reviewing the appropriate engineering drawing for a system component or section in Appendix A of the Trial Burn Plan.

Note: Engineering projects are underway for design, purchase, and installation of stack gas CEMS and upgrade of the waste feed automatic shutoff system.

4.2 <u>Incinerator Operating Conditions</u>

Table 4-2 presents a summary of the incinerator's process operating data for each test run. Where appropriate, coefficients of variation are noted for each process parameter, to give an indication of the variabilities recorded during testing periods. In general, process variability was steady as indicated by CV's under $\pm 10\%$.

Figures 4-2 through 4-11 present the Provox computerized output of the waste feed rate for Test Runs 1-1 through 3-3 and including the additional HC/Cl₂ sample runs on 2/13/89 for Test Runs 1-1 and 1-2. The top graph indicates actual waste feed rate; the bottom graph indicates change in flow rate between consecutive signal averaging times. Test Runs 1-1 through 1-3 show the miscalibrated span for the Signet waste flow meter; the actual waste flow rate values were post-calibrated to be 0.371 times that shown for Test Condition 1. The inadvertent waste feed shutoff during Test Run 1-1 is graphically depicted in Figure 4-2 with a return to steady feed rate at about 1155 hrs.

Table 4-3 presents a summary of CAC Residue feed rate descriptive statistics, based on the Provox output which logs recordings every 30 seconds.

Table 4-4 presents a summary of the calculated heat input to the incinerator for each test run. Natural gas auxiliary fuel usage was minimized during the trial burn, as is normal operation for the incinerator. No gas pressure gauge was installed during Test Condition 1, so heat input from natural gas was estimated based on an assumed line pressure. Heat input from natural gas was graphically calculated from a vendor-supplied burner curve for the John Zink Model HI-24 gas burner and a heat content of 1100 Btu/scf gas.

TABLE 4-1. SUMMARY OF CAC INCINERATOR PROCESS MONITORS

Parameter	Location of monitor ^a	Type of monitor	Operating range	Units recorded
CAC residue feed rate	1	Magnetic flowmeter	0-2000	lb/hr
Azo residue feed rate	2	Mass flowmeter	0-600	lb/hr
Atomizing steam	3	Pressure gauge	0-110	psig
Auxiliary fuel gas flow rate	4	Pressure gauge	0-50	psig
Combustion air flow rate	5	Anemometer	0-5100	fpm
Burner flame	6	UV detector		flame failure
Thermal oxidizer temperature (4	•	Thermocouple (Type K)	500-1500	°C
Quench H ₂ O flow rate	8	Magnetic flowmeter	0-100	gpm
Quench pot temperature (3	9	Thermocouple (Type J)	0-200	°c
Scrubber recycle flow rate		Magnetic flowmeter	0-100	gpm
Scrubber/absorbe inlet gas temp		Thermocouple (Type J)	0-200	°c
Scrubber water flow rate	11	Magnetic flowmeter	0-200	gpm
Scrubber/absorbe water level	r 12	DP cell	0-100	8
Utility water boosted pressu	13	Pressure gauge	0-200	psig
Oxygen (to be installed)	14	Thermomagnetic	0-21	%
Carbon Monoxide	15	Extractive	0-100	ppm
(to be install	ed)	NDIR	0-2000	ppm
Utility water flow rate (to be install	17	Magnetic flow meter	0-300	gpm
(CO DE INSCAIL				

a Refer to Figure 4-1.

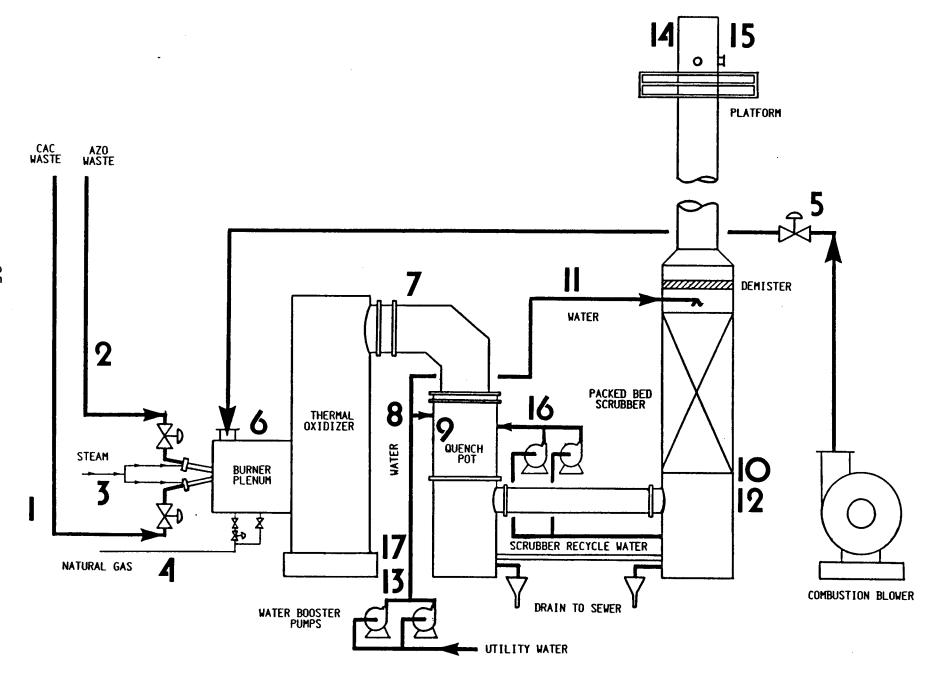


Figure 4-1. Location of CAC Incinerator System Process Control Sensors and Monitors.

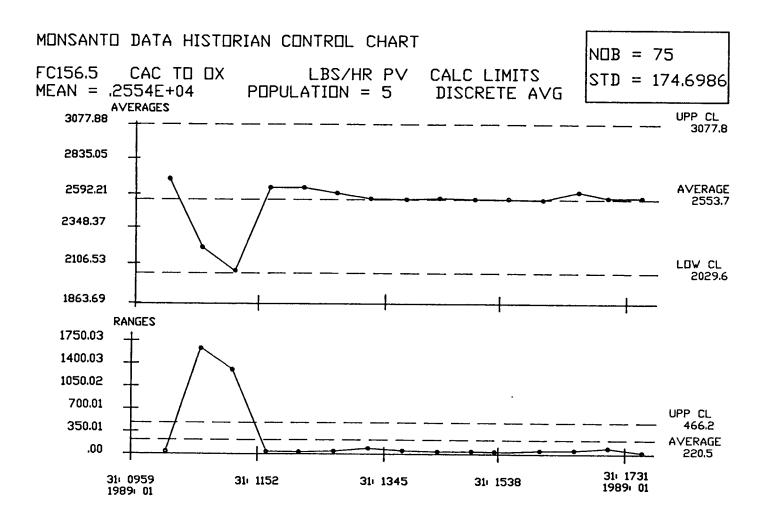


Figure 4-2. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 1-1, 31-Jan-89.

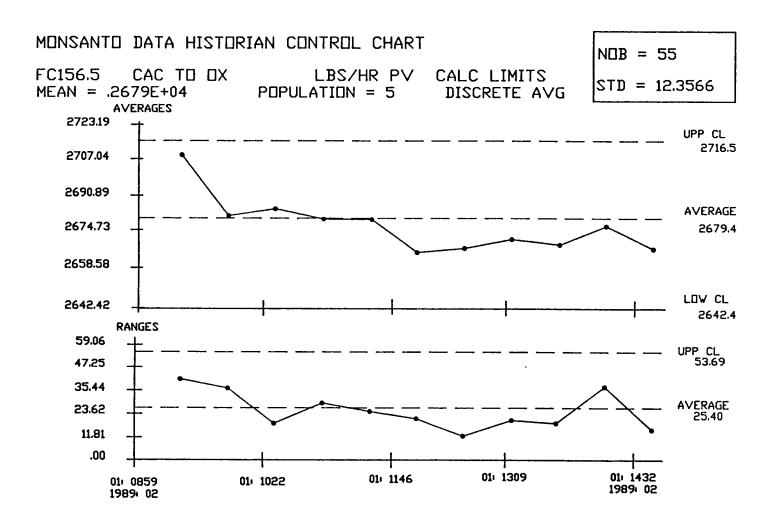


Figure 4-3. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 1-2, 1-Feb-89.

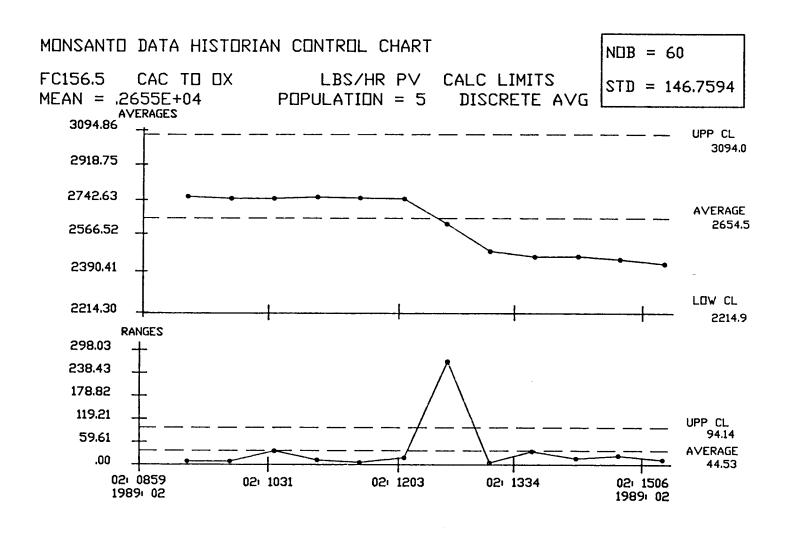


Figure 4-4. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 1-3, 2-Feb-89.

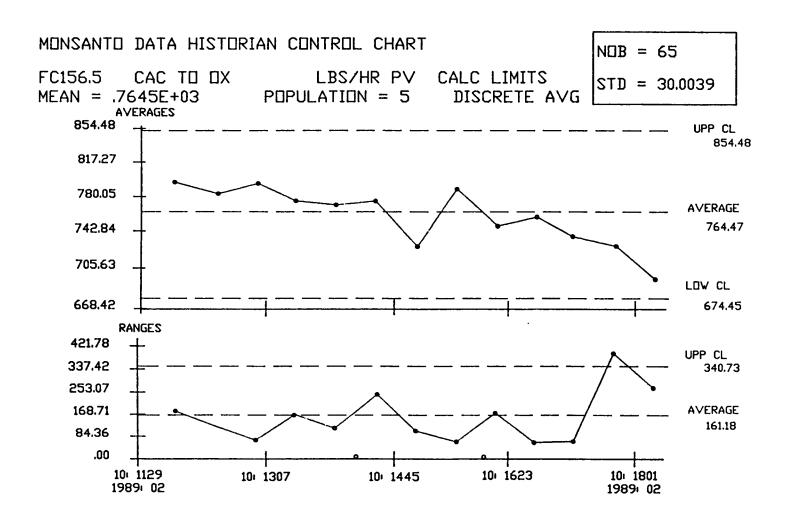


Figure 4-5. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 2-1, 10-Feb-89.

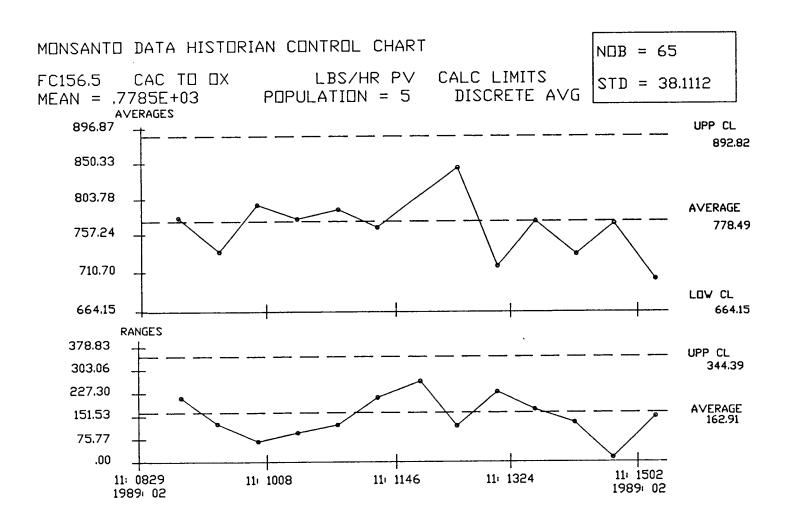


Figure 4-6. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 2-2, 11-Feb-89.

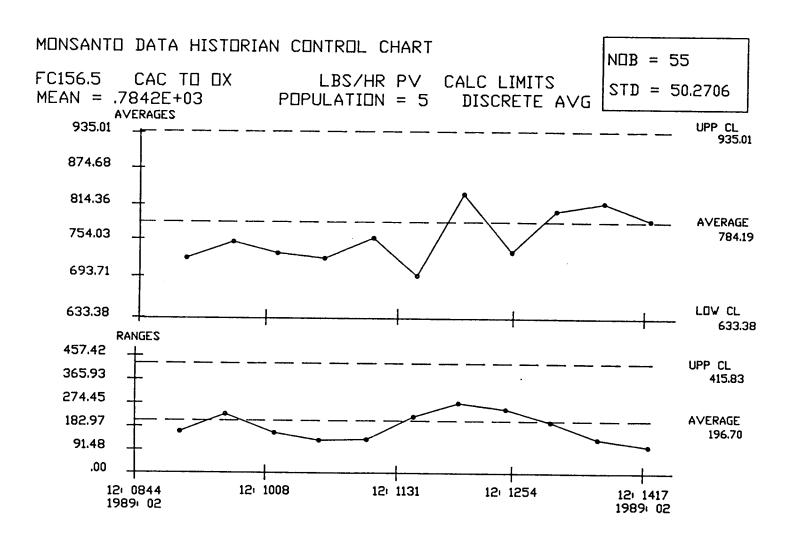


Figure 4-7. CAC Residue Feed Rate to Incinerator System, Monsanto-Queeny Plant, Test Run 2-3, 12-Feb-89.

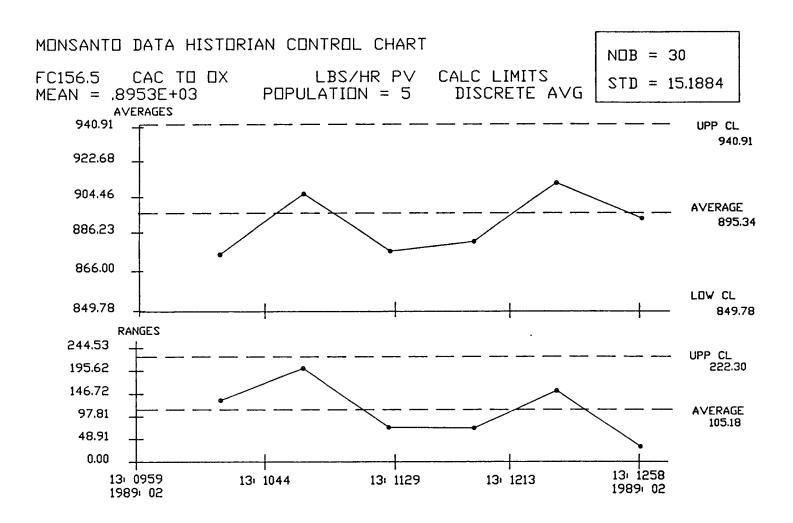


Figure 4-8. CAC Residue Feed Rate to Incinerator System, Monsanto-Queeny Plant, Test Run HCl/Cl₂ for 1-1 and 1-2, 13-Feb-89.

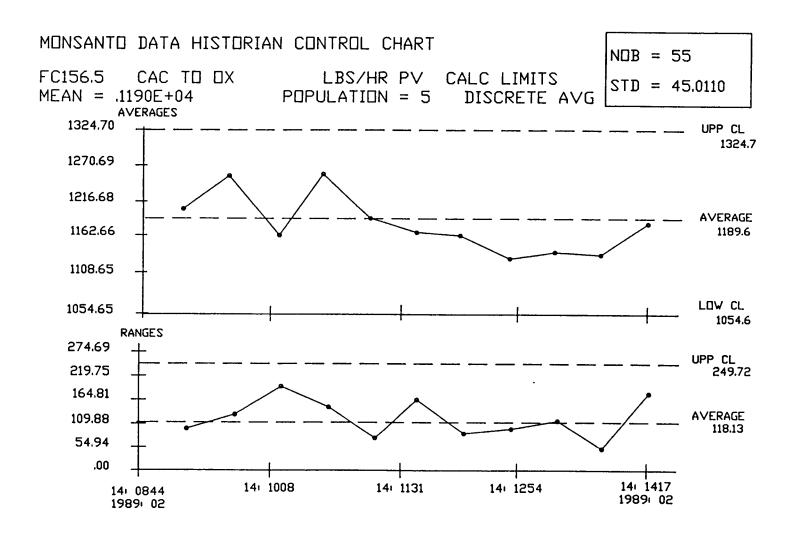


Figure 4-9. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 3-1, 14-Feb-89.

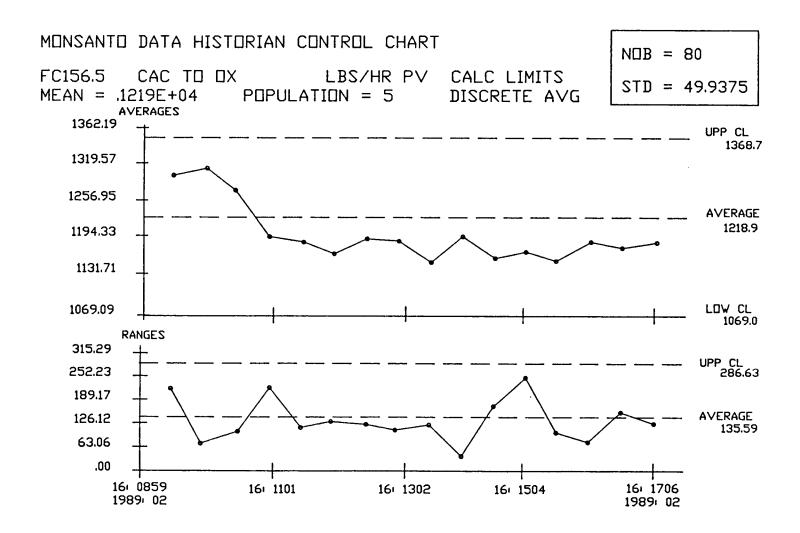


Figure 4-10. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 3-2, 16-Feb-89.

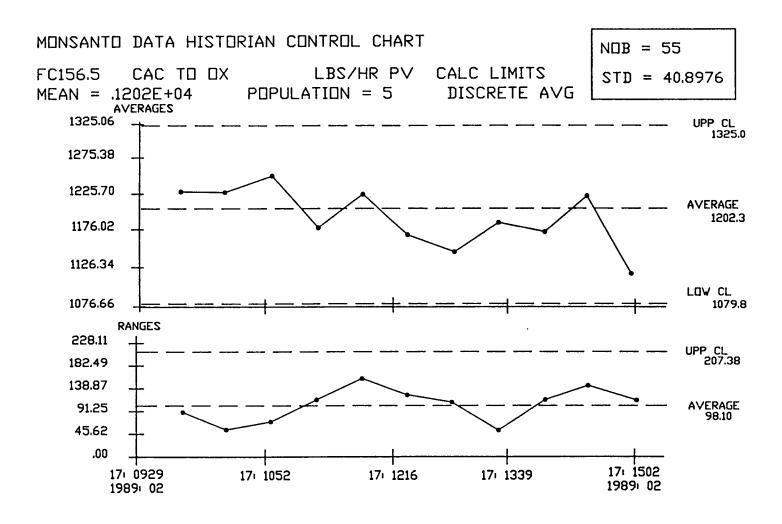


Figure 4-11. CAC Residue Feed Rate to Incinerator System,
Monsanto-Queeny Plant, Test Run 3-3, 17-Feb-89.

		Run Number							
Parameter	1-1	1-2	1-3	2-1	2-2	2-3	3–1	3-2	3–3
CAC Waste Feed Rate, lb/hr									
Avg of 1/2-hr readings	1012	1056	1034	706	772	800	1202	1224	1215
CV	8.2%	0.3%	5.7%	11.6%	1.8%	5.3%	7.5%	3.6%	2.7%
Provox Avg for run	947.4	994.1	984.8	764.5	778.5	895.3	1189.6	1218.9	1202.3
CV	6.8%	0.5%	5.5%	3.9%	4.9%	1.7%	3.8%	4.1%	3.4%
Natural Gas Pressure, psig				8.5	9.0	9.0	8.9	8.3	8.9
Atomizing Steam, psig	110	110	108	112	113	113	112	113	114
Combustion Air Flow, ft/min	2039	2077	2129	1975	1964	1962	2235	2264	2264
CV	3.6%	0.8%	1.1%	1.9%	1.6%	1.4%	1.2%	0.8%	1.0%
Combustion Air Flow, acfm	1601	1631	1671	1550	1542	1540	1754	1779	1777
Oxidizer Temperature, C	1025	1049	1034	985	988	992	1068	1056	1062
CV	3.1%	0.5%	1.5%	0.8%	0.6%	1.3%	0.6%	0.7%	0.4%
Quench Water Flow, gpm	48.5	47.9	47.8	49.1	49.3	50.6	49.3	51.3	54.0
	5.2%	1.3%	1.3%	7.2%	7.3%	9.4%	7.3%	8.8%	8.7%
Scrubber Water Flow, gpm	205.1	205.1	205.1	205.1	205.1	205.1	205.1	205.1	205.1
Recycle Water Flow, gpm	46.5	42.6	40.6	44.9	45.5	45.1	46.3	45.4	45.1
	10.8%	2.3%	4.2%	2.1%	1.8%	1.7%	1.1%	1.7%	1.5%
Scrubber Water Level, %	78	76	76	77	77	77	76	76	76
Quench Outlet Temperature, C	76.5	79.9	80.4	75.2	75.6	75.6	81.6	82.9	83.7
·	6.0%	4.5%	4.6%	4.0%	3.4%	4.1%	15.7%	5.2%	7.3%
Scrubber Inlet Temperature, C	33.9	37.9	38.9	26.7	26.2	26.2	39.1	39.9	40.0
•	9.7%	1.1%	4.1%	1.1%	1.9%	2.3%	2.6%	2.4%	2.5%
Quench Effluent Flow, gpm	96.1	95.6	101.3	107.6	101.0	100.8	103.6	110.8	100.9
Scrubber Effluent Flow, gpm	66.4	81.0	82.0	81.0	81.7	79.7	84.3	85.0	83.3

CV = Coefficient of Variation = (std dev/mean) X 100%

TABLE 4-3. SUMMARY OF WASTE FEED RATE DESCRIPTIVE STATISTICS

_	Waste		within Test	
Run	Feed Rate	Number of	Standard	Coefficient
۱o.	lb/hr	Recordings	Deviation	of Variation
1-1	947.4	900	64.8	6.8%
1-2	994.1	660	4.48	0.5%
1-3	984.8	720	54.45	5.5%
Avg	975.4			
CV	2.1%			
2-1	764.5	780	30.0	3.9%
2-2	778.5	780	38.11	4.9%
2-3	895.3	480	15.19	1.7%
Avg	812.8			
CV	7.2%			
3-1	1189.6	660	45.01	3.8%
3-2	1218.9	960	49.94	4.1%
3-3	1202.3	660	40.90	3.4%
Avg	1203.6			
CV	1.0%			

TABLE 4-4. SUMMARY OF HEAT INPUT TO CAC INCINERATOR FOR EACH TEST RUN

Run No.	Gas Press psig	Gas Burner Heat Input Btu/hr	CAC Waste Feed Rate lb/hr	CAC Waste Btu/lb	CAC Waste Heat Input mmBtu/hr	Total Input mmBtu/hr
1-1	9.0 ^a	870,000	947	5,740	5.436	6.306
1-2	9.0 ^a	870,000	994	5,763	5.728	6.598
1-3	9.0 ^a	870,000	985	5,725	5.639	6.509
2-1	8.5	800,000	765	5,730	4.383	5.183
2-2	9.0	870,000	779	5,860	4.565	5.435
2-3	9.0	870,000	895	5,805	5.195	6.065
3-1	8.9	850,000	1190	5,750	6.842	7.693
3-2	8.3	750,000	1219	5,665	6.906	7.656
3-3	8.9	850,000	1202	5,465	6.569	7.419

^aAssumed values; no pressure gauge installed during test

4.3 Air Pollution Control Equipment Operating Conditions

Table 4-2 presented the entire process operating conditions during each test run. Details of the process logs during the trial burn can be found in Appendix H -- Process Data Sheets. Quench, scrubber and recycle water flow rates were recorded every 30 minutes during testing from the CRT output of the Provox control system. Quench outlet temperature is an average of three thermocouple readings in the quench section, and scrubber inlet temperature is a gas temperature reading taken just below the scrubber packing. Quench and scrubber effluent flow rates were measured once per test run using a graduated plastic drum and timer. Within each Test Run and within each Test Condition, variability was low for APCE parameters, indicative of steady operation.

5. SAMPLING AND ANALYSIS RESULTS

This section presents all of the sampling and analysis results obtained during the trial burn. Regulatory emission performance results have been previously displayed and discussed in Section 3.

5.1 <u>Methods Description</u>

Figure 5-1 illustrates the sampling locations at the CAC Incinerator. Table 5-1 presents a summary of the entire trial burn program's sampling and analytical methods. Detailed procedures are given in Appendix I -- Sampling and Analysis Methods.

The liquid CAC Residue was sampled from a sampling valve located near the point where the waste feed entered the burner, as shown in Figure 5-2. The valve outlet was equipped with a septum from which waste samples were withdrawn into a 150-mL glass syringe. A 100-mL aliquot of CAC Residue was collected every 30 minutes during each test run period. Each aliquot was added to a 1-gallon amber glass bottle for compositing. At the conclusion of each test run, the contents of the composite bottle were mixed and the following aliquots were poured out for subsequent analysis:

One 40-mL vial for POHC analysis
One 40-mL vial for total organic halide (TOX) analysis
One 500-mL glass bottle for metals analysis
One 250-mL bottle for elemental, ash and Btu analysis

In between test run periods, tetrachloroethene (PCE) was added to the CAC Residue storage tank by drum pump into the recycle line return to tank. This was done to spike the CAC Residue to up a 2.0 wt% PCE concentration, with confirmational analysis of PCE concentration by the plant laboratory. The added PCE was allowed to mix via line recirculation (tank turnover every 15-20 minutes) before PCE spike level confirmation.

Samples of utility feed water were collected from a tap on the booster pumps' discharge line. Samples of the quench and scrubber effluent waters were collected from valved discharge lines from the quench and packed column scrubber. plastic drum and timer were used once each test run to measure the quench and scrubber effluent flow rate. Each water stream was sampled at 30 minute intervals throughout each test run period. A 200-mL grab sample was collected and compositied into a 1-gallon amber glass bottle. At the end of each test run, the composite was mixed; an aliquot was taken for TOX analysis into a 250-mL amber bottle and an aliquot for total chloride, suspended solids, and dissolved solids analyses was taken in to 500-mL polyethylene bottle. In addition, grab samples at the beginning, middle, and end of each test run were collected in 40-mL vials for VOA POHC analysis.

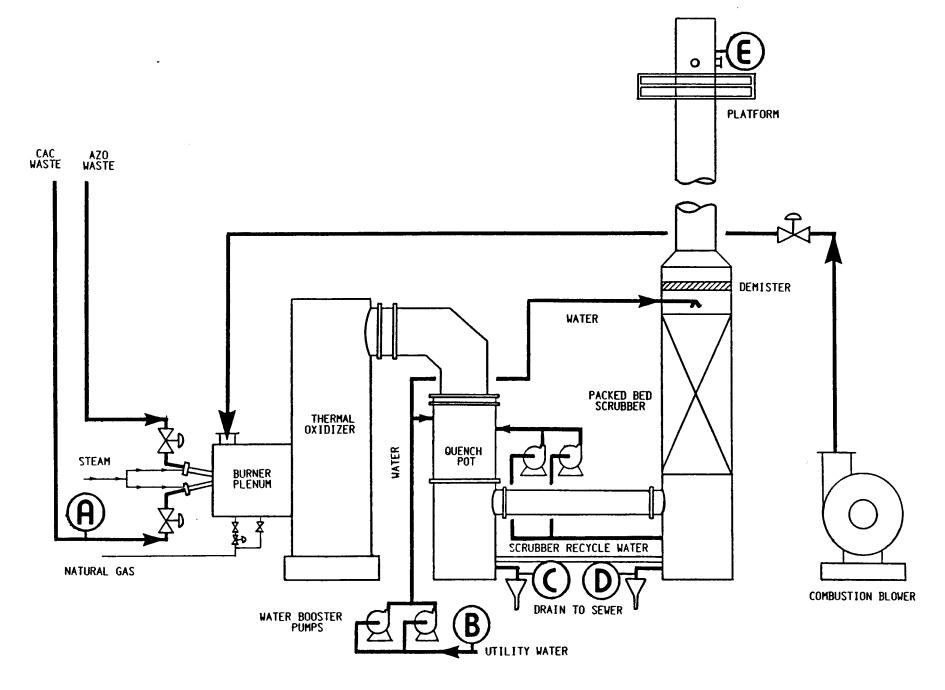


Figure 5-1. Schematic Diagram of Sampling Locations, CAC Incinerator System, Monsanto-Queeny Plant.

TABLE 5-1. SUMMARY OF SAMPLING AND ANALYTICAL METHODS

Sample	Sample procedure	No. of samples	Analytical parameters	Analytical procedures
CAC waste feed	5004 (Tap)	One grab/30 minutes; composited into one 250-mL amber and one	POHCs	EPA 3580; EPA 8240 solvent dilution and direct injection GC/MS
		500-mL clear glass. Three VOA grabs/run.	Metals	EPA 3050; EPA 6010; EPA 7060 (Pb); EPA 7421 (As); EPA 7470 (Hg) acid digestion then
			Btu content	ICP or GFAAS or CVAAS ASTM D240 bomb calorimetry
			Moisture	Karl-Fischer titration
			Ash	ASTM D482 gravimetric loss on
			Ultimate	ignition RSTM D3176
			analysis (C,H,N,O,S,C1)	calorimetry, absorption
			Organochlorine	EPA 3580; EPA 9020 dilution and micro-
			Viscosity	coulometry ASTM D445 viscometer
Utility water	5004 (Tap)	One grab/30 minutes; composited into one 250-mL amber and one	POHCs	EPA 5030; EPA 8240 purge & trap, packed column GC/MS
		500-mL clear glass. Three VOR grabs/run.	ρΗ	EPA 9040 electrometry at sample collection
			Total chloride	EPA 300.0 ion chromatography
			Organochlorine (TOX)	EPA 9020 microcoulometry
			Suspended solids	EPA 160.1 filter, filtrate evapor-
			Dissolved	ation, and gravimetric EPA 160.2
			solids	filter, residue desicc- ation, and gravimetric

TABLE 5-1. SUMMARY OF SAMPLING AND ANALYTICAL METHODS (Continued)

Sample	Sample procedure	No. of samples	Analytical parameters	Analytical procedures
Quench effluent	5004 (Tap)	One grab/30 minutes; composited into one 250-mL amber and one 500-mL clear glass. Three VOR grabs/run.	POHCs pH	EPA 5030; EPA 8240 purge & trap, packed column GC/MS EPA 9040 electrometry at samp collection
			Total chloride	EPA 300.0
			Organochlorine (TOX) Suspended solids Dissolved solids	microcoulometry EPA 160.1 filter, filtrate eva ation, and gravimetr EPA 160.2 filter, residue des:
Scrubber effluent	5004 (Tap)	One grab/30 minutes;	POHCs	ation, and gravimeto EPA 5030; EPA 8240
ocrabber en raent	Joby (Tap)	composited into one 250-mL amber and one 500-mL clear glass. Three VOA grabs/run.	рН	purge & trap, packed column GC/MS EPR 9040 electrometry at samp
		mi ee von gi abs/i an.	Total chloride	collection
			Organochlorine	ion chromatography EPA 9020
			(TOX) Suspended solids	microcoulometry EPA 160.1 filter, filtrate ev
			Dissolved solids	ation, and gravimet EPR 160.2 filter, residue des ation, and gravimet
Particulate emissions	EPA Method 5	One 2-hr traverse per test run; 60 ft gas sample minimum	Particulates	EPA Method 5 desiccation and gravimetric
Dry gas molecular weight	EPA Method 3	One 2-hr traverse per test run; probe attached to EPA 5 probe; 70-L gas bag	CO2, O2	EPA Method 3 Orsat analysis

N

TABLE 5-1. SUMMARY OF SAMPLING AND ANALYTICAL METHODS (Continued)

Sample	Sample procedure	No. of samples	Analytical parameters	Analytical procedures
HCl emissions	EPA Method 5 back half uses 0.1 N NaOH soln	One 2-hr traverse per test run; 60 ft gas sample minimum	Total chlorides	EPA 300.0 ion chromatography
HC1/C12 emissions	Draft EPA Method using Greenburg- Smith impingers	One 1-hr traverse per test run; 60 ft gas sample minimum	HCl, Cl2	EPA 300.0 ion chromatography
Volatile POHC and PIC emissions	EPA Method 0030 (VOST)	Three 20-L gas samples per test run	POHCs, PICs	EPA 5040; EPA 8240 thermal desorption, packed column GC/MS
Semivolatile POHC and PIC emissions	EPA Method 0010 (Semi-VOST)	One 3-hr traverse per test run; 105 ft gas sample minimum	POHCs, PICs	EPA 3510; EPA 3540; EPA 8270 separatory funnel and Soxhlet extractions, capillary column GC/MS
Carbon monoxide emissions	EPA Method 10 (extractive)	Continuous	Carbon monoxide	EPA Method 10 filter, desiccate, and continuous NDIR
Stack gas oxygen	EPA Method 3A (extractive)	Continuous	0×ygen	EPA Method 3A filter, desiccate, and continuous ZrO2 electro- chemical
Fugitive emissions	EPR Method 21 (OVA)	One time screen survey	Fugitive VOCs	EPA Method 21 portable FID instrument

POHCs = Principle Organic Hazardous Constituents GC/MS = Gas Chromatography/Mass Spectroscopy VOA = Volatile Organic Analysis (40-mL vial)

PICs = Products of Incomplete Combustion

ICP = Inductively Coupled Plasma Atomic Absorption Spectroscopy

GFAAS = Graphite Furnace Atomic Absorption Spectroscopy

CVAAS = Cold Vapor Atomic Absorption Spectroscopy

TOX = Total Organic Halides

VOST = Volatile Organic Sampling Train NDIR = Nondispersive Infrared Spectrometer ZrO2 = Zirconium Oxide Electrochemical Cell

OVA = Organic Vapor Analyzer FID = Flame Ionization Detector

ASTM = American Society for Testing and Materials

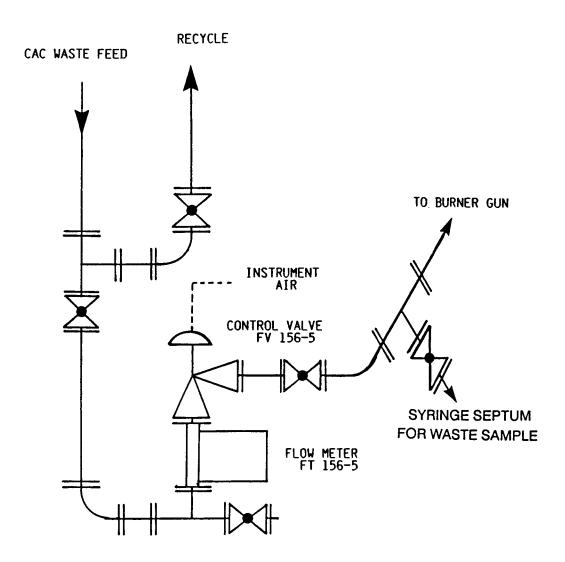


Figure 5-2. CAC Residue Sampling Location.

Process operation parameters were also recorded every 30 minutes during each test run period. The Provox computer control system logs three parameters every 30 seconds (waste feed rate, oxidizer temperature, and combustion air velocity), with CRT screen output for several others. The following is a summary of the process parameters recorded and their output locations:

CAC Residue feed rate	
Auxiliary fuel	
Atomizing steam	Steam pressure gauge
Combustion air velocity	
Oxidizer temperature	Provox screen #2
Quench temperatures (3)	
Quench water flow	
Scrubber inlet temperature	Provox screen #2
Scrubber water flow	Provox screen #2
Scrubber recycle water flow	Provox screen #2
Scrubber water level	Control room strip chart
Utility water pH	Measured with each sample
Quench effluent pH	Measured with each sample
Scrubber effluent pH	Measured with each sample

The stack sampling location is shown in more detail in Figure 5-3. Sampling ports were located 17.0 feet above the neck-down at the packed column scrubber exit and 5.0 feet below the 26-inch I.D. stack exit. Two sample ports at a 90° angle were used for particulate, HCl, and semivolatile organic (MM5) sampling. A twelve point traverse was selected according to EPA Method 1 procedures, and is shown in Figure 5-3. A third sample port located between the other two was angled down at approximately 45° and used for the volatile organic sampling train (VOST) and the CEMS probes.

The sampling train used for particulates and total chlorides is a modification of EPA Method 5, in which the back-half or impinger portion is modified to include an absorbing solution for chlorides, as shown in Figure 5-4. The first three impingers had 150-mL each of 0.2N sodium carbonate (Na₂CO₃) solution, followed by an empty impinger to catch any carryover, and then a final impinger containing silica gel for drying the sample gas.

A different sampling train was used for separate collection of hydrogen chloride (HCl) and chlorine gas (Cl₂), as shown in Figure 5-5. This sampling train used a heated probe packed with glass wool to remove particulates, three Greenburg-Smith impingers with 200-mL acidic solution (0.1N $\rm H_2SO_4$) to selectively absorb HCl and allow Cl₂ to pass through, and then two impingers containing 200-mL alkaline absorbing solution (0.1N $\rm Na_2CO_3$) to convert Cl₂ to chloride for chloride ion analysis by ion chromatography (EPA Method 300.0). Figure 5-6 illustrates the flow diagram for chloride ion analysis.

POHC (1,2-DCE and PCE) and volatile PIC emissions were sampled using the VOST (EPA Method 0030). Analysis of the sorbent traps was by thermal desorption (EPA Method 5040) and packed column GC/MS (EPA Method 8240) set on wide-scan. Figure 5-7 illustrates the thermal desorption method procedure and Figure 5-8 illustrates the volatiles GC/MS procedure.

Semivolatile POHC (PCE) and PIC emissions were sampled using the Modified Method 5 (EPA Method 0010) train, which uses a sorbent resin of XAD-2 after the particulate filter/oven to trap semivolatile organic compounds. Figure 5-9 illustrates the semivolatile organic sampling train; Figure 5-10 illustrates the sampling train's recovery and cleanup procedure. Preparation for analysis of the recovered liquids is by separatory funnel liquid-liquid extraction and concentration of the extract. Preparation of the recovered solids is by Soxhlet extraction and Both final prepared extracts are combined for concentration. analysis, as shown in Figure 5-11. Figure 5-12 illustrates the preparation procedure for separatory funnel extraction (EPA Method 3510), and Figure 5-13 illustrates the the preparation procedure for Soxhlet extraction (EPA Method 3540). Semivolatile organic analysis was done using capillary column GC/MS (EPA Method 8270), as shown in Figure 5-14. The CAC Residue was diluted before analysis using EPA Method 3580, shown in Figure 5-15.

E POINT LOCATION		STACK		
DISTANCE IN STACK, inches		SAMPLING PORTS		5 ft
1.1		00	H	+
3.8		•	ĺ	
7.7	:			
18.3				
22.2				17 ft
24.9				
		SCRUBBER		
	DISTANCE IN STACK, inches 1.1 3.8 7.7 18.3 22.2	1.1 3.8 7.7 18.3 22.2 24.9	DISTANCE IN STACK, inches 1.1 3.8 7.7 18.3 22.2 24.9 PACKED TOWER SCRUBBER	DISTANCE IN STACK, inches 1.1 3.8 7.7 18.3 22.2 24.9 PACKED TOWER

Figure 5-3. CAC Incinerator Stack Sampling Location and Traverse Points.

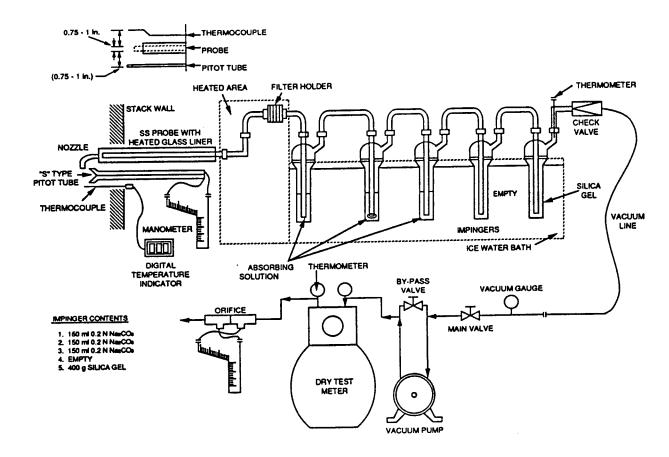


Figure 5-4. Schematic Diagram of EPA Method 5 Sampling Train for Particulate and Total Chloride.

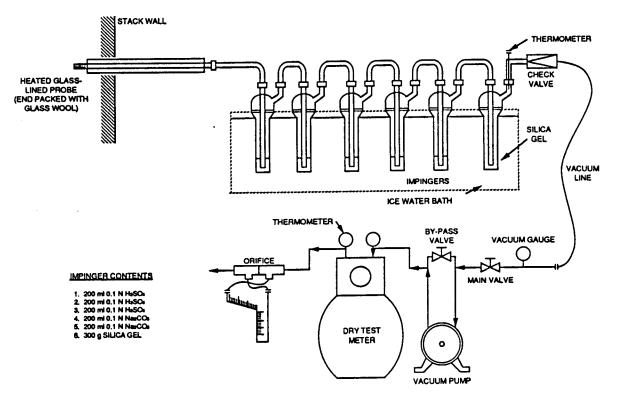


Figure 5-5. Schematic Diagram of HCl/Cl₂ Sampling Train.

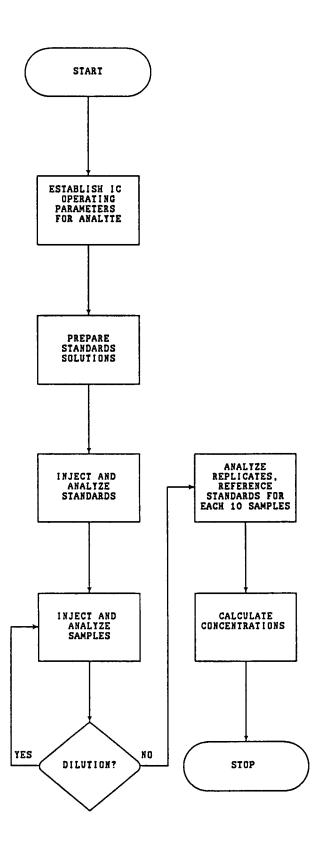


Figure 5-6. Flow Diagram for Chloride Ion Analysis (EPA Method 300.0)

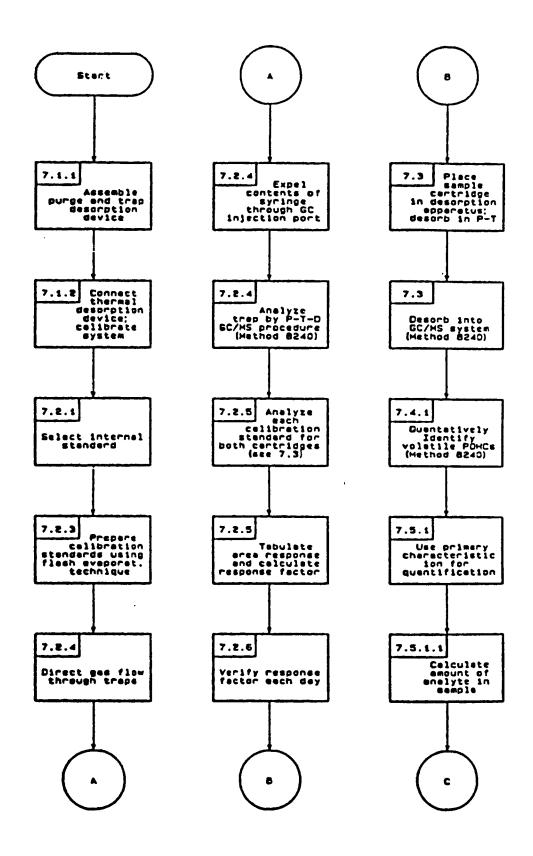


Figure 5-7. Protocol for Analysis of Sorbent Cartridges from VOST (EPA Method 5040).

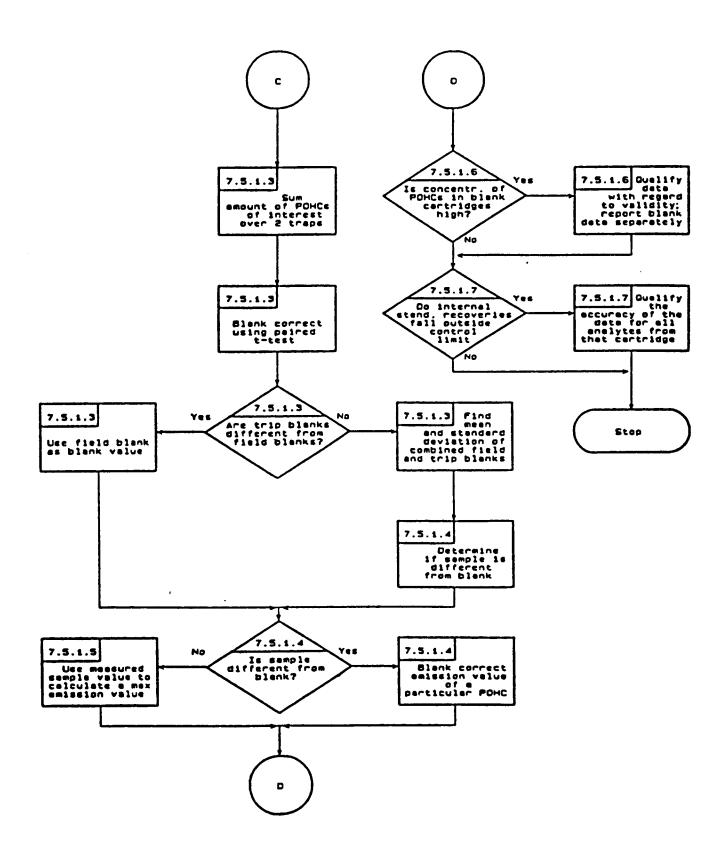


Figure 5-7. Protocol for Analysis of Sorbent Cartridges from VOST (EPA Method 5040). (Continued)

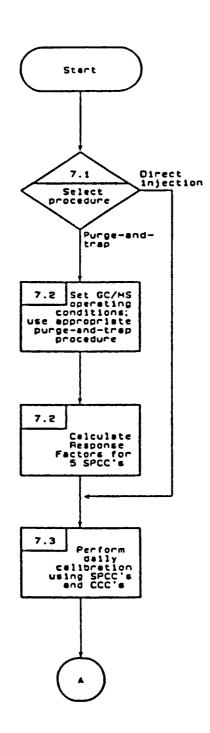


Figure 5-8. Protocol for Packed Column GC/MS for Volatile Organics (EPA Method 8240).

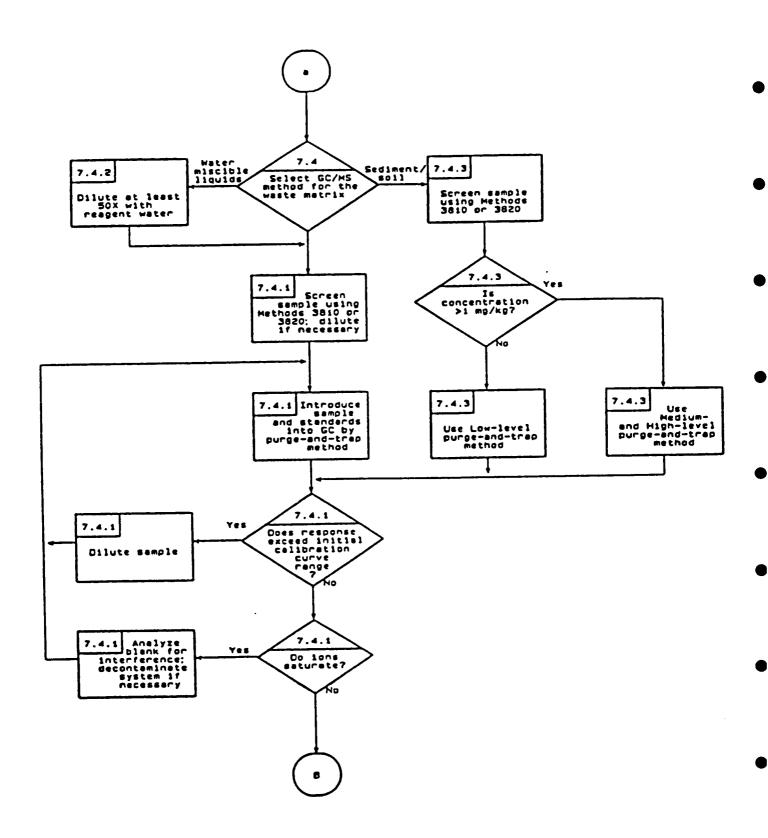


Figure 5-8. Protocol for Packed Column GC/MS for Volatile Organics (EPA Method 8240).

(Continued)

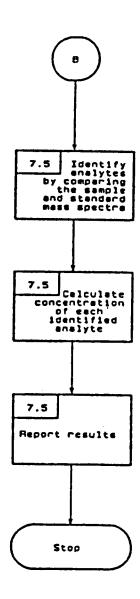


Figure 5-8. Protocol for Packed Column GC/MS for Volatile Organics (EPA Method 8240). (Continued)

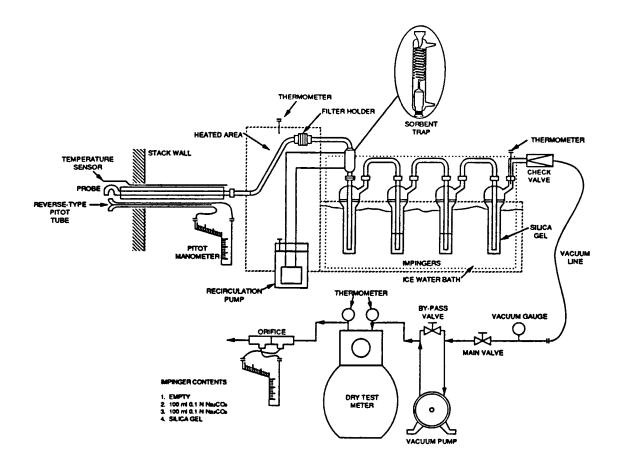


Figure 5-9. Schematic Diagram of Semivolatile Organic Sampling Train (EPA Method 0010).

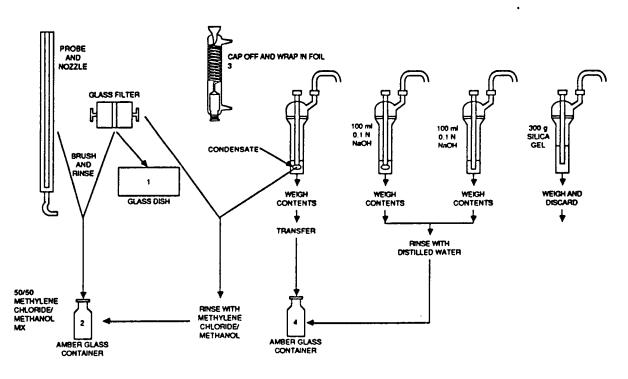


Figure 5-10. Sample Recovery Flow Diagram for Semivolatile Organic Sampling Train.

FILTER SOXHLET SPIKE WITH EXTRACT IN SURROGATES MeCb XAD-2 PROBE RINSE SPIKE WITH CONCENTRATE TO 1 ml MeOH/MeCk **SURROGATES** COMBINED CONDENSATE/ ACID/BASE LIQUID EXTRACTION NaOH FRACTION CONCENTRATE GC/MS ANALYSIS TO 1 ml

Figure 5-11. Flow Chart for Semivolatile Analytical Preparation.

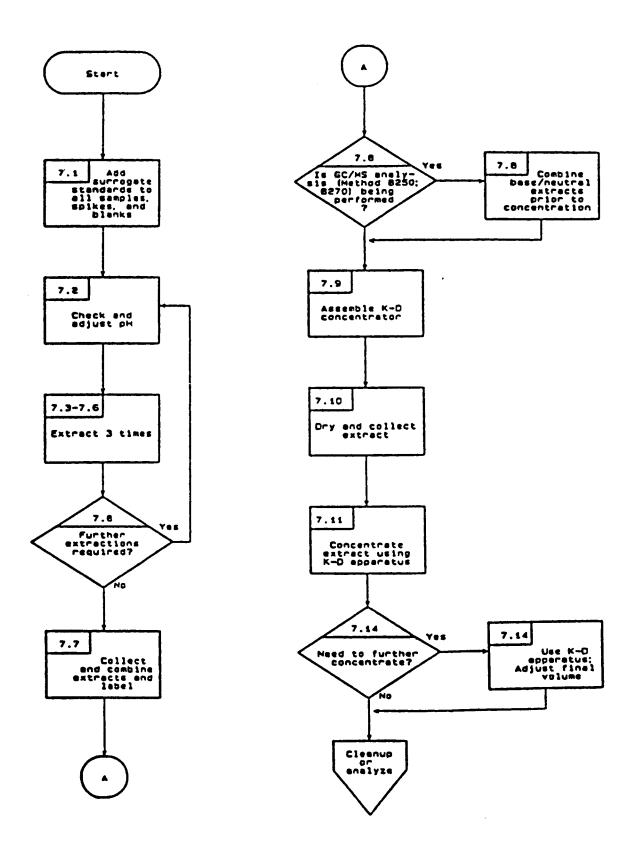


Figure 5-12. Protocol for Separatory Funnel Liquid-Liquid Extraction (EPA Method 3510).

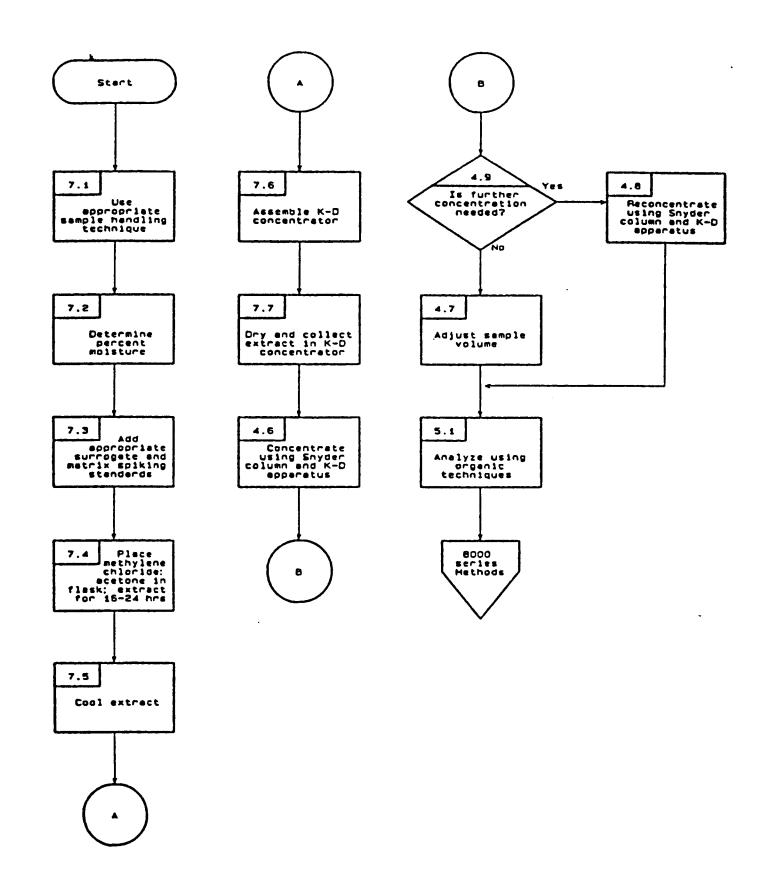


Figure 5-13. Protocol for Soxhlet Extraction of Semivolatile Organics (EPA Method 3540).

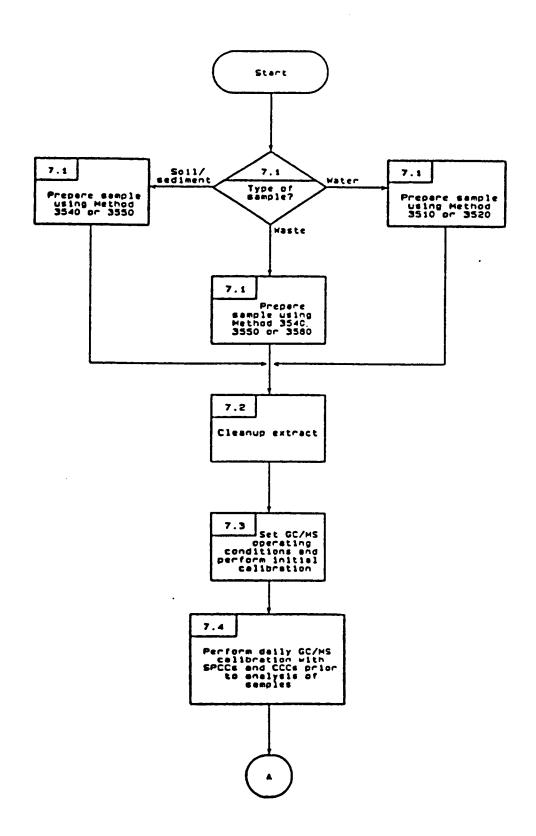


Figure 5-14. Protocol for Capillary Column GC/MS for Semivolatile Organics (EPA Method 8270).

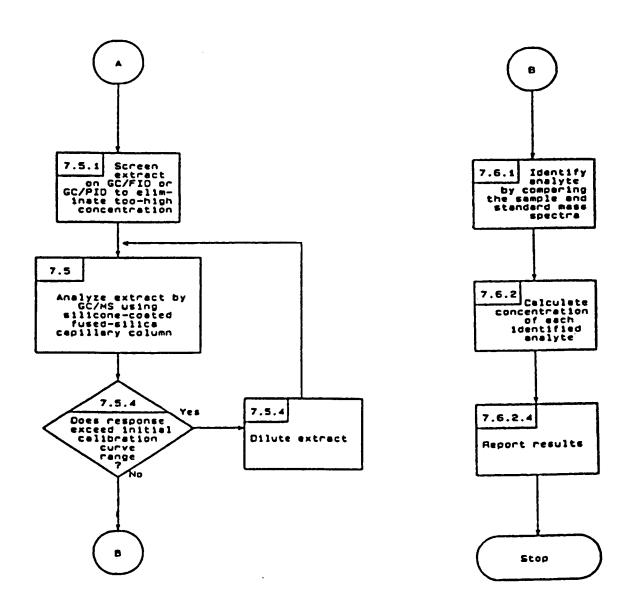


Figure 5-14. Protocol for Capillary Column GC/MS for Semivolatile Organics (EPA Method 8270). (Continued)

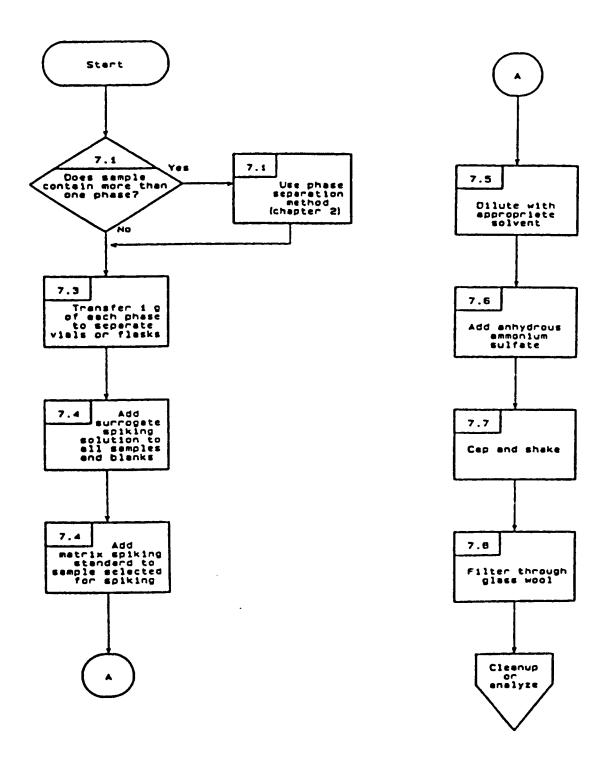


Figure 5-15. Protocol for Waste Dilution Before Analysis (EPA Method 3580)

The CEM sampling system for CO and O_2 in the stack gas is illustrated schematically in Figure 5-16. It consisted of an instack particulate filter followed by a gas stream condenser in ice bath to cool and dry the sample gas before entering the continuous analyzers (EPA Method 10 for CO and EPA Method 3A for O_2). A three-way valve before the condenser allowed for calibration gas introduction through the entire sample line. CEMS analyzer output was recorded on an Okagawa digital/analog strip chart recorder. Appendix I -- Sampling and Analysis Methods provides detailed descriptions of the procedures used for all stack gas sampling and for chloride analysis. Analysis procedures for waste, waters, and gas samples are contained in EPA methods manuals (EPA-SW-846 and EPA-600/4-84-017).

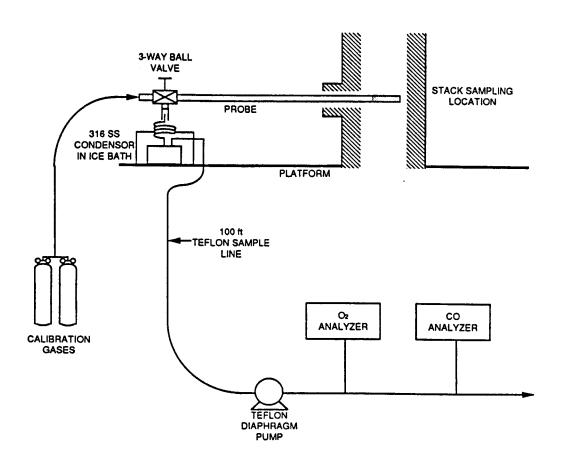


Figure 5-16. Diagram of CEM Sampling System for CO and O_2 .

5.2 Waste Feed and Fuel Characteristics

The CAC Residue was characterized for each test run for numerous parameters related to its incinerability and the resulting emissions. Table 5-2 presents a summary of the CAC Residue characterization data. Most characterization parameters remained constant from day-to-day, as indicated by the low coefficients of variation. Levels of 1,2-dichloroethane fluctuated from 0.33% to 1.14% (3300 ppm to 11,400 ppm) as the CAC process changed over the nearly three week period encompassing the trial burn test. This variability in DCE concentration is not unusual. The trial burn plan to hold constant at 2.0% the concentration of tetrachloroethene (PCE) was well met as PCE concentrations ranged from 1.69% to 2.40% with a CV of only ± 9.9 %. Waste density and chloride content remained extremely steady with CV's of ± 0.4 % and ± 2.9 %, respectively.

TOX analyses were a wasted effort on the CAC Residue because the analytical procedure is just not applicable to highly chlorinated liquid wastes. At least million-fold dilutions were required to bring the TOX concentration into the analytical instrument's operating range. EPA Method 9020 protocol requires duplicate analyses for each measurement, indicative of a potentially imprecise procedure. EPA Method 9020 is applicable to TOX determinations when trace chlorinated compounds exist in waters.

Ash concentrations were quite low, ranging from 0.03% to 0.07%. No water was found in the waste, an expected result since the CAC Residue is water-reactive (D003). Water analysis had to be changed to the Karl-Fischer titration procedure; the ASTM D95 xylene codistillation procedure included a QC water spike to determine percent recovery, which was impossible due the waste's reactivity with water.

Concentrations of metals were also trivial or nonexistent. Arsenic, beryllium, cadmium, silver and thallium were all not detected. Barium was detected in five of nine samples, just above the detection limit (0.046 mg/L). A trace of mercury was detected in one sample, barely above the detection limit (0.0012 mg/L) and probably an analytical artifact. Chromium and lead were found in nearly all waste samples, with chromium ranging from 0.53 to 10.5 mg/L and lead from below detection (<0.018 mg/L) to 0.61 mg/L. Chromium and lead in the CAC Residue are most likely from vessel and pipeline corrosion where the process and waste stream are exposed to metallic alloy surfaces.

5.3 Stack Gas Concentration Data

Table 5-3 presents a summary of the CAC Incinerator exhaust gas flow rate and composition data. Included are data from both the particulate (designated by a "5") and the semivolatile ("M5") test runs, which spanned most of the testing period. Within each

TABLE 5-2. SUMMARY OF CAC RESIDUE CHARACTERIZATION DATA, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Parameter	Units	1-1	1-2	1-3	2-1	Run No. 2-2	2-3	3-1	3-2	3-3	Avg	CV
1,2-Dichloroethane	wt %	0.33	0.42	0.33	0.69	0.64	0.81	1.14	1.07	1.05	0.72	41.9%
Tetrachloroethene	wt %	2.01	2.40	2.38	2.22	2.06	2.31	1.69	2.02	2.09	2.13	9.9%
Heat Value	Btu/lb	5,740	5,763	5,725	5,730	5,860	5,805	5,750	5,665	5,465	5,723	1.8%
Water Content	wt %	<0.02	< 0.02	<0.02	<0.02	< 0.02	< 0.02	<0.02	< 0.02	< 0.02	<0.02	
Ash	wt %	0.05	0.05	0.05	0.07	0.06	0.04	0.06	0.03	0.05	0.05	21.5%
Viscosity @ 19 C	cps	4.4	4.3	4.4	4.4	4.4	4.4	4.3	4.4	4.4	4.4	0.9%
Density @ 19 C	g/mL	1.291	1.290	1.289	1.283	1.279	1.277	1.283	1.282	1.285	1.284	0.4%
Total Chlorides	wt %	41.44	42.06	41.96	44.40	40.15	40.00	42.19	41.26	41.37	41.65	2.9%
Carbon	wt %	NA	33.25	NA	NA	33.18	NA	NA	30.58	NA	32.34	3.8%
Hydrogen	wt %	NA	2.50	NA	NA	4.04	NA	NA	3.17	NA	3.24	19.5%
Oxygen	wt %	NA	22.05	NA	NA	22.57	NA	NA	24.98	NA	23.20	5.5%
Nitrogen	wt %	NA	<0.01	NA	NA	<0.01	NA	NA	< 0.01	NA	<0.01	
Sulfur	wt %	NA	<0.01	NA	NA	< 0.01	NA	NA	<0.01	NA	<0.01	
Total Organic Halogen	g/L	471	358	122	123	180	132	141	45.3	31.7	178	76.4%
(by EPA Method 9020)	wt %	36.48	27.75	9.46	9.59	14.07	10.34	10.99	3.53	2.47	13.85	76.1%
Antimony (Sb)	mg/L	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	<1.55	
Arsenic (As)	mg/L	< 0.030	<0.030	<0.030	<0.030	<0.030	< 0.030	< 0.030	< 0.030	<0.030	< 0.030	
Barium (Ba)	mg/L	< 0.046	<0.046	< 0.046	0.060	0.054	0.072	0.076	< 0.046	0.054	0.056	
Beryllium (Be)	mg/L	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	<0.018	
Cadmium (Cd)	mg/L	< 0.054	< 0.054	<0.054	<0.054	<0.054	<0.054	< 0.054	< 0.054	<0.054	<0.054	
Chromium (Cr)	mg/L	0.53	0.60	0.65	10.5	0.69	0.74	8.35	3.41	1.39	2.98	120%
Lead (Pb)	mg/L	0.074	0.066	0.22	0.61	0.074	0.60	0.38	<0.018	<0.018	0.23	
Mercury (Hg)	mg/L	<0.0012	< 0.0014	0.0022	<0.0012	<0.0012	<0.0012	<0.0012	<0.0012	< 0.0012	0.0013	
Silver (Ag)	mg/L	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	<0.08	
Thallium (TI)	mg/L	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	

NA = Not analyzed

2	Test No. 5-1-1 M5-1-1 5-1-2 M5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2 5-2-3	Temp oF 51 51 50 50 49 49 50.0 1.6%	14.5 13.9 14.0 13.9 13.5 14.2 14 2.2%	Flow acfm 3198 3069 3086 3074 2976 3145 3091 2.2% 2788 2756	3221 3053 3135 3137 3071 3248	1.26 1.30 1.20 1.19 1.16 1.16 1.21 4.3%	10.5 10.5 10.5 9.9 10.0 10.6 10.25 2.8%	8.6 8.9 9.4 9.1 9.0 8.4 8.9 3.7%	N2 % 80.9 80.6 80.7 80.9 81.0 81.0	96.7 97.4 86.8 87.8 98.3 92.5 5.40
2	5-1-1 M5-1-1 5-1-2 M5-1-2 5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	51 50 50 49 49 50.0 1.6%	14.5 13.9 14.0 13.9 13.5 14.2 14 2.2%	3198 3069 3086 3074 2976 3145 3091 2.2%	3221 3053 3135 3137 3071 3248 3144 2.3%	% 1.26 1.30 1.20 1.19 1.16 1.16 1.21 4.3%	% 10.5 10.5 9.9 10.0 10.0 10.6 10.25 2.8%	8.6 8.9 9.4 9.1 9.0 8.4 8.9 3.7%	% 80.9 80.6 80.7 80.9 81.0 81.0	96.7 97.4 86.8 88.0 87.8 98.3
2	M5-1-1 5-1-2 M5-1-2 5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	51 50 50 49 49 50.0 1.6%	13.9 14.0 13.9 13.5 14.2 14 2.2%	3069 3086 3074 2976 3145 3091 2.2%	3053 3135 3137 3071 3248 3144 2.3%	1.30 1.20 1.19 1.16 1.16 1.21 4.3%	10.5 9.9 10.0 10.0 10.6 10.25 2.8%	8.9 9.4 9.1 9.0 8.4 8.9 3.7%	80.6 80.7 80.9 81.0 81.0 80.9 0.2%	97.4 86.8 88.0 87.8 98.3 92.5 5.40
2	5-1-2 M5-1-2 5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	50 49 49 50.0 1.6%	14.0 13.9 13.5 14.2 14 2.2%	3086 3074 2976 3145 3091 2.2%	3135 3137 3071 3248 3144 2.3%	1.20 1.19 1.16 1.16 1.21 4.3%	9.9 10.0 10.0 10.6 10.25 2.8%	8.9 9.4 9.1 9.0 8.4 8.9 3.7%	80.6 80.7 80.9 81.0 81.0 80.9 0.2%	97.4 86.8 88.0 87.8 98.3 92.5 5.40
2	M5-1-2 5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	50 49 49 50.0 1.6%	13.9 13.5 14.2 14 2.2% 12.6 12.5	3074 2976 3145 3091 2.2%	3137 3071 3248 3144 2.3%	1.19 1.16 1.16 1.21 4.3%	10.0 10.0 10.6 10.25 2.8%	9.1 9.0 8.4 8.9 3.7%	80.9 81.0 81.0 80.9 0.2%	92.5 5.4°
2	5-1-3 M5-1-3 Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	49 49 50.0 1.6%	13.5 14.2 14 2.2% 12.6 12.5	2976 3145 3091 2.2%	3071 3248 3144 2.3%	1.16 1.16 1.21 4.3%	10.0 10.6 10.25 2.8%	9.1 9.0 8.4 8.9 3.7%	80.9 81.0 81.0 80.9 0.2%	98.3 98.3 92.5 5.49
2	Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	50.0 1.6%	14.2 14 2.2% 12.6 12.5	3145 3091 2.2% 2788	3248 3144 2.3% 2912	1.16 1.21 4.3% 0.97	10.6 10.25 2.8%	8.4 8.9 3.7%	81.0 81.0 80.9 0.2%	92.5 5.4 ⁹
2 M	Avg CV 5-2-1 M5-2-1 5-2-2 M5-2-2	50.0 1.6% 44 44	14 2.2% 12.6 12.5	3091 2.2% 2788	3144 2.3% 2912	1.21 4.3% 0.97	10.25 2.8%	8.9 3.7%	80.9 0.2%	98.3 92.5 5.49
N	CV 5-2-1 M5-2-1 5-2-2 M5-2-2	1.6% 44 44	2.2% 12.6 12.5	2.2% 2788	2.3% 2912	4.3% 0.97	2.8%	3.7%	0.2%	5.49
N	5–2–1 M5–2–1 5–2–2 M5–2–2	44 44	12.6 12.5	2788	2912	0.97				
N	M5-2-1 5-2-2 M5-2-2	44	12.5				10.9	7.9	81.2	103.4
٨	5-2-2 M5-2-2				2900					
٨	M5-2-2	44	40.5			0.96	10.8	8.0	81.2	101.5
			12.5	2762	2894	0.94	10.9	8.0	81.1	103.7
	5-2-2	44	12.9	2859	3000	0.95	10.8	8.0	81.2	101.5
	J-2-J	43	12.5	2765	2906	0.92	10.7	8.1	81.2	99.7
	M5-2-3	43	12.9	2844	3004	0.91	11.0	7.8	81.2	105.4
	Avg	43.7	13	2796	2936	0.94	10.85	8.0	81.2	102.
	CV	1.1%	1.4%	1.5%	1.6%	2.2%	0.9%	1.2%	0.0%	1.8
3	5-3-1	44	14.0	3105	3268	0.95	9.7	9.3	81.0	83.0
٨	M5-3-1	44	14.5	3205	3375	0.94	9.6	9.4	81.0	81.5
:	5-3-2	43	13.8	3062	3273	0.92	9.8	9.0	81.2	84.2
M	M5-3-2	44	13.9	3064	3273	0.93	10.0	8.8	81.2	87.4
	5-3-3	43	13.8	3052	3260	0.92	9.6	9.2	81.2	81.1
M	M5-3-3	43	14.3	3165	3388	0.91	9.2	9.6	81.2	75.2
	Avg CV	43.5 1.1%	14.1 1.9%	3109 1.9%	3306	0.92 1.4%	9.65	9.2	81.1	82.1

Test Condition, all stack gas parameters such as temperature, velocity, volumetric flow rate, and percents of $\rm H_2O$, $\rm O_2$, and $\rm CO_2$ remained very constant, as indicated by the extremely low CV's.

5.3.1 <u>POHCs/DRE</u> --

Destruction and removal efficiency of the two POHCs chosen for this trial burn -- 1,2 dichloroethane (DCE) and tetrachloroethene (PCE) -- was measured using the VOST and packed column GC/MS. Also, an attempt to measure DRE of PCE was made using the semivolatile organic sampling train and capillary column GC/MS.

DRE results were presented in Table 3-1 along with POHC feed rates and emission rates. Table 3-2 presents the CAC Residue POHC analysis results; Table 3-3 presents the calculation of POHC feed rates (W_{in}); and Table 3-4 presents calculations of POHC emission rates (W_{out}). DRE of 1,2-dichloroethane averaged emission rates (Wout). DRE of 1,2-dichloroethane averaged 99.9999% for Test Conditions 1 and 3 (Normal and High) and was >99.9999% ((not detected in the stack gas) for Test Condition 2 (Low). DRE of tetrachloroethene averaged 99.9997% for Test Condition 1, >99.9999% for Test Condition 2, and 99.9999% for Test Condition 3. The high DREs for tetrachloroethene demonstrate that the incinerator is able to effectively destroy difficult-to-incinerate organic compounds. Recent studies at the University of Dayton Research Institute (UDRI) revealed that high chloride concentrations in a waste tend to stabilize tetrachloroethene; however, the CAC Incinerator was able to achieve five and six "nines" DRE with a 40% Cl waste feed.

Use of the semivolatile organic sampling train with XAD-2 resin for determining tetrachloroethene emissions was a failure, as predicted by the author and the testing contractor. Percent recoveries from spiked XAD-2 sorbent traps for PCE were too low to be valid, ranging from 30-38%. An analytical PCE standard was run using EPA Method 8270, and the percent recovery was 91%. Clearly, XAD-2 sorbent and the desorption procedure of EPA Method 0100 is inappropriate for this compound. It may be possible to increase the sorbent recovery using cold solvent desorption, but this technique is not part of the standard method procedure. The following response was provided to Missouri DNR and EPA Region VII during the trial burn plan comment period:

"Both I and the S&A contractor (PEI Associates, Inc.) agree that requirement of an additional set of runs using the Modified Method 5 (EPA Method 0010) for tetrachloroethylene is unnecessary The sorbent retention volume for a particular and wasteful. compound demonstrates a semilog-linear relationship with boiling temperature. Because the boiling tetrachloroethylene is near the lower bound of usefulness for XAD-2 sorbent, the potential for POHC sample loss by volume breakthrough is high. This is not the case with Tenax-GC, used Moreover, the VOST uses a backup sorbent cartridge in the VOST. containing part charcoal to protect against breakthrough, whereas

the MM5 train does not use a backup sorbent cartridge. Also, XAD-2 must be solvent desorbed and then concentrated before analysis, both of which are contributors to further POHC sample losses. The VOST will collect, retain, and submit higher concentrations to the analytical instrument than the MM5 for tetrachloroethylene. I suggest that the commenter contact Dr. Larry D. Johnson, USEPA/EMSL in RTP, NC at 919/541-7943 for guidance on the use of appropriate sorbents in stack sampling. I see no need to "spike" an additional semivolatile POHC into the waste when the MM5 is not to be used and when the waste stream is primarily comprised of volatile compounds."

5.3.2 PICs --

Table 5-4 presents a summary of the PIC emission rates for those compounds which were detected in the stack gas. PIC emissions were measured in this trial burn by conducting a widescan analysis of the volatile (VOST) and semivolatile (MM5) gas samples collected. A full wide-scan analysis of PICs is provided in Appendix A -- Example Calculations.

The CAC Incinerator uses chlorinated city water as the process water for quench and scrubber. Several common drinking water contaminants were observed in the stack gas as the pollutants became air-stripped by the hot exhaust gas from the quench/scrubber system, such as chloroform, bromoform, bromodichloromethane, and dibromochloromethane. Chloroform is also likely to be a PIC from combustion of chlorinated ethanes and ethenes. The relative percentage of city water contaminants appearing as stack gas PICs ranged from 7-64% throughout the trial burn, with the relative percentage decreasing during the high waste feed rate test condition. Several volatile compounds also appeared in the blank VOST tubes, with chloroform in two of the field blanks. Acetone appeared in nearly all blanks, ranging from 17-190 ng per pair. Other periodic appearances in VOST blanks were made by chloromethane, methylene chloride, bromodichloromethane, dibromochloromethane, hexanone, styrene, chloride, xylenes, chlorobenzene, and benzene, all at quite small concentrations.

The most common semivolatile organic PIC was benzoic acid. Total volatile PIC emissions (including air-stripped city water pollutants) ranged from 0.96 to 10.17 grams per hour, with the highest emissions occurring during Test Condition 3. Total semivolatile PIC emissions ranged from 1.09 to 2.40 grams per hour.

TABLE 5-4. SUMMARY OF PIC EMISSION RATES, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

그 사람들 가장 그는 그를 가장했다.				Ro	ın Number					
	1-1	1-2	1-3	2-1	2-2	2-3	3-1	3-2	3–3	
Compound	lb/hr	ib/hr	łb/hr	lb/hr	ib/hr	lb/hr	lb/hr	lb/hr	lb/hr	Comments
VOLATILES										
Chloromethane	3.71E-04	3.21E-04	4.95E-04	2.92E-03	1.88E-03	4.30E-03	2.02E-02	1.21E-02	1.00E-02	
Bromomethane	4.72E-06	7.38E-06	7.41E-06	ND	ND	4.87E-05	ND	9.72E-05		City water pollutant
Chloroethane	1.14E-05	1.62E-05	2.00E-05	7.14E-06	ND	ND	1.34E-05	1.97E-05	1.48E-05	City water politicant
Methylene Chloride	1.68E-05	9.26E-05	2.95E-05	2.51E-05	2.40E-05	3.24E-05	3.36E-05	3.67E-05		Lab contaminant or P
Acetone	9.78E-05	1.02E-04	9.54E-05	8.13E-05	9.21E-05	1.32E-04	9.17E-05	9.24E05	6.21E-05	
Carbon Disulfide	4.41E-06	1.17E-05	1.09E-05	3.59E-05	2.57E-05	1.80E-05	ND	7.86E-06	ND	Lab contaminant or Fi
1.1-Dichloroethene	3.38E-06	3.42E-06	3.82E-06	ND ND	ND	ND	4.78E-06	6.75E-06	ND	Con Communicati
1,1-Dichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	
trans-1,2-Dichloroethene	2.36E-06	3.14E-06	ND	ND	ND	ND	ND	5.23E-06	ND	
Chloroform	9.44E-04	7.95E-04	8.65E-04	7.13E-04	7.25E-04	7.10E-04	1.03E-03	1.05E-03	9.74E-04	City water and/or PIC
2-Butanone	ND	ND	ND	6.00E-06	1.02E-05	8.73E-06	1.34E-05	9.07E-06	8.25E-06	Ony water and/or FIG
1,1,1-Trichloroethane	4.11E-06	5.40E-06	1.02E-05	8.75E-06	8.26E-06	1.05E-05	1.07E-05	5.81E-06	6.95E-06	
Carbon Tetrachloride	2.74E-04	3.06E-04	3.23E-04	1.82E-04	1.81E-04	2.42E-04	4.48E-04	5.27E-04	4.26E-04	
Bromodichloromethane	3.25E-04	2.55E-04	2.63E-04	2.82E-04	3.00E-04	3.02E-04	3.24E-04	2.94E-04		City water pollutant
Trichloroethene	2.21E-06	4.85E-06	4.37E-06	ND	ND	2.84E-06	2.86E-06	3.82E-06	3.56E-06	Ony water politicant
Dibromochloromethane	1.24E-04	9.93E-05	9.85E-05	9.57E-05	1.03E-04	1.01E-04	1.14E-04	1.12E-04		City water pollutant
Benzene	5.44E-06	1.56E-05	1.86E-05	1.49E-05	1.20E-05	5.81E-06	2.47E-05	1.46E-05	9.96E-06	Ony water policiant
Bromoform	3.84E-05	3.17E-05	4.58E-05	1.60E-05	1.62E-05	1.65E-05	1.01E-04	2.77E-05		City water pollutant
Toluene	1.92E-06	2.40E-05	3.30E-05	3.40E-06	2.36E-06	ND	4.00E-06	5.11E-06	8.83E-06	Only water policialit
Chlorobenzene	5.30E-06	8.85E-06	1.27E-05	1.60E-05	1.08E-05	1.10E-05	1.03E-05	1.08E-05	5.63E-06	
Ethylbenzene	ND	2.56E-06	ND	ND	ND	ND	ND	ND	8.63E-06 ND	
Total Xylenes	4.90E-06	9.42E-06	1.00E-05	4.05E-06	ND	ND	ND	ND	4.12E-06	_
Volatiles (Ib/hr) =	0.00224	0.00212	0.00235	0.00234	0.00339	0.00594	0.02243	0.01443	0.01207	-
Volatiles (g/hr) =	1.02	0.96	1.06	1.08	1.54	2.69	10.17	6.54	5.48	
City H2O Vols (%) =	64.1%	56.2%	54.5%	47.3%	33.7%	19.8%	7.0%	11.0%	12.4%	
SEMIVOLATILES										
1.4-Dichlorobenzene	ND	ND	ND	ND	1.76E-05	1.79E-05	ND	ND	ND	
Benzoic Acid	1.55E-03	1.28E-03	2.20E-03	2.68E-03	3.03E-03	1.99E-03	2.76E-03	2.83E-03	2.25E-03	
Di-n-butyl phthalate	ND	1.30E-05	ND	ND	1.76E-05	2.86E-05	1.79E-05	2.51E-05		Lab contaminant
ois(2-Ethylhexyl)phthalate	7.03E-05	7.29E-05	9.57E-05	3.52E-05	7.40E-05	3.94E-04	5.37E-05	ND		Lab contaminant
lon-App XIII constituents	2.57E-03	2.76E-03	2.99E-03	NA NA	NA NA	NA NA	NA NA	NA NA	NA	Las Comaninant
Semivolatiles (lb/hr) =	0.00419	0.00413	0.00529	0.00272	0.00314	0.00243	0.00283	0.00286	0.00239	:
Semivolatiles (g/hr) =	1.90	1.87	2.40	1.23	1.42	1.10	1.28	1.29	1.09	

ND - Not detected

NA = Not analyzed

5.3.3 Carbon Monoxide --

Carbon monoxide concentration in the stack gas was measured continuously throughout the trial burn testing period, in conjunction with continuously measured oxygen concentration. Table 5-5 presents a summary of the CO and O_2 continuous emission monitoring data. CO concentrations through out the trial burn remained below detection (<5 ppm), indicative of steady process operation. CO concentration remained below 10 ppm even during the inadvertent waste feed shutoff in Test Run 1-1. O_2 concentrations fluctuated slightly with small changes in combustion air and waste feed rate, but also remained steady with no abrupt changes. CEMS O_2 concentrations correlated well with integrated bag Orsat determinations. Field data strip charts for the CEMS can be found in Appendix E -- Field Data Sheets.

5.3.4 Particulate Matter --

Particulate emission results were summarized in Table 3-8, shown earlier in the report. Details of the calculations used can be found in Appendix A - Example Calculations. Field data sheets of the particulate runs can be found in Appendix E - Field Data Sheets, and gravimetric analysis results can be found in Appendix F - Analytical Reports.

Table 5-6 presents a summary of theoretical particulate emission control efficiencies for the quench/scrubber system, based on the assumption of 100% conversion of ash in the waste to particulate. Control efficiencies ranging from 97.9 to 99.0% can be estimated on the above basis.

5.3.4 <u>Hydrogen Chloride</u> --

Total chlorides were measured using the alkaline absorbing solution of the back-half of the EPA Method 5 train and ion chromatography. Speciation of HCl and ${\rm Cl}_2$ emissions were measured using a draft EPA method for this purpose, contained in Appendix I -- Sampling and Analysis Methods. Tables 3-6 and 3-7 presented the total chloride, HCl, and ${\rm Cl}_2$ emission results and the incineration system removal efficiencies for HCl.

TABLE 5-5. SUMMARY OF CONTINUOUS EMISSION MONITORING DATA

	Run	Time	Carbon Mo	noxide, ppm	Oxygen, %
Date	No.	Period	Avg ^a	Range	Avg Range
					
1/31	1-1	0930-1730	<5	<5 - <5	10.1 8.9-13.5
2/1	1-2	0930-1421	<5	<5 - <5	9.5 9.3-9.6
2/2	1-3 _b	0850-1502	<5	<5 - <5	10.0 9.6-10.4
2/13	_D	1015-1300	<5	<5 - <5	10.5 10.4-10.6
2/10	2-1	1115-1745	<5	<5 - <5	10.8 10.5-12.9
2/11	2-2	0855-1438	<5	<5 - <5	10.8 10.6-11.0
2/12	2-3	0845 - 1428 ^C	<5	<5 - <5	10.9 10.0-11.3
2/14	3-1	0845-1400	<5	<5 - <5	12.6 11.8-13.3
2/16	3-2	0915-1715	<5	<5 - <5	10.2 8.7-10.9
2/17	3-3	0930-1507	<5	<5 - <5	9.7 9.4-10.1

^aAverage of 60-minute rolling averages. ^bExtra test runs for HCl/Cl₂. ^COxygen analyzer off calibration at 1315 hours.

TABLE 5-6. SUMMARY OF THEORETICAL PARTICULATE EMISSION CONTROL EFFICIENCIES, CAC INCINERATOR, MONSANTO CHEMICAL COMPANY, J. F. QUEENY PLANT

Test Cond.	Run No.	Waste Feed Rate lb/hr	Ash %	Theoret. Control	Partic Emissions lb/hr	Theor Max Ash lb/hr
1 Normal	5-1-1 5-1-2	947.4 994.1	0.0005 0.0005	97.9% 98.5%	0.0102 0.0074	0.474 0.497
HOLMAL	5-1-3	984.8	0.0005	98.5%	0.0075	0.492
	Avg CV		0.0005	98.3%	0.0084 15.4%	0.488
2 Low	5-2-1 5-2-2 5-2-3	764.5 778.5 895.3	0.0007 0.0006 0.0004	99.0% 98.6% 98.1%	0.0053 0.0067 0.0069	0.535 0.467 0.358
	CV Avg		0.0006	98.6% 0.4%	0.0063 11.3%	0.453 16.1%
3 High	5-3-1 5-3-2 5-3-3	1189.6 1218.9 1202.3	0.0006 0.0003 0.0005	98.5% 97.3% 98.2%	0.0107 0.0099 0.0109	0.714 0.366 0.601
	Avg CV		0.0005 26.7%	98.0% 0.5%	0.0105 4.2%	0.560 25.9%

5.4 APCE Aqueous Streams

Utility water into the quench/scrubber sections of the CAC Incinerator and effluents from the quench and scrubber were sampled and analyzed for POHCs, pH, total chlorides, TOX, and suspended and dissolved solids. Neither of the POHCs were detected (MDL = 3-5 ug/L) in any of the aqueous samples. Table 5-7 presents a summary of the total chloride, TOX, and solids data for the aqueous streams. Most chlorides exit the incinerator system via the quench effluent, as also indicated by the water pH results, shown in Table 5-8. Quench and scrubber effluent pH remained constant during the trial burn tests, with no buildup of acidic components. A similar trend is seen in the aqueous TOX results. A slight increase in solids was observed for the quench effluent, but no increase was observed for scrubber effluent concentrations.

5.5 VHAP Fugitive Emissions Screening

Volatile hazardous air pollutants (VHAPs) were emission screened at the incinerator system piping from the waste feed pump up to the burner gun. No reading higher than 7 ppm as CH_4 was observed for any process component; ambient background levels were 5 ppm. The field data sheet for the VHAP emissions screening can be found in Appendix E.

TABLE 5-7. SUMMARY OF WATER INFLUENT/EFFLUENT ANALYSIS RESULTS AT CAC INCINERATOR, MONSANTO-QUEENY PLANT

Sample Stream	Run No.	Total Chloride mg/L	Suspended Solids mg/L	Dissolved Solids mg/L	Total Organic Halogen mg/L
Utility	U-1-1	32.9	3	305	0.144
Feed	U-1-2 U-1-3	30.9 30.1	<1 3	283 264	0.160 0.028
	Avg	31.3	2	284	0.111
	CV	3.8%	NA	5.9%	53.1%
	U-2-1	48.4	<1	264	0.132
	U-2-2	30.3	<1	272	0.109
	U-2-3	34.2	<1	296	0.120
	Avg	37.6	<1	277	0.120
	CV	20.7%	NA	4.9%	7.8%
	U-3-1	31.8	<1	279	0.079
	U-3-2	32.7	<1	267	0.143
	U-3-3	30.3	<1	264	0.172
	Avg	31.6	<1	270	0.131
	CV	3.1%	NA	2.4%	29.6%
Quench	QE-1-1	8000	<1	318	0.774
Effluent	QE-1-2	7160	<1	322	0.455
	QE-1-3	7130	<1	298	0.448
	Avg	7430	<1	313	0.559
	CV	5.4%	NA	3.4%	27.2%
	QE-2-1	4930	<1	297	0.361
	QE-2-2	5160	<1	306	0.455
	QE-2-3	4518	<1	333	0.721
	Avg	4869	<1	312	0.512
	CV	5.5%	NA	4.9%	29.8%
	QE-3-1	7860	<1	311	0.855
	QE-3-2	8700	<1	298	0.707
	QE-3-3	9270	<1	292	0.960
	Avg	8610	<1	300	0.841
	CV	6.7%	NA	2.6%	12.3%

TABLE 5-7. SUMMARY OF WATER INFLUENT/EFFLUENT ANALYSIS RESULTS AT CAC INCINERATOR, MONSANTO-QUEENY PLANT (Continued)

Sample Stream	Run No.	Total Chloride mg/L	Suspended Solids mg/L	Dissolved Solids mg/L	Total Organic Halogen mg/L
Scrubber	SE-1-1	1140	<1	270	0.508
Effluent	SE-1-2	612	<1	284	0.531
	SE-1-3	3550	<1	301	0.389
	Avg	1767	<1	285	0.476
	CV	72.4%	NA	4.4%	13.1%
	SE-2-1	212	<1	282	0.128
	SE-2-2	249	<1	289	0.357
	SE-2-3	251	<1	315	0.148
	Avg	237	<1	295	0.211
	CV	7.6%	NA	4.8%	49.1%
	SE-3-1	546	<1	282	0.350
	SE-3-2	533	<1	267	0.452
	SE-3-3	670	<1	272	0.468
	Avg	583	<1	274	0.423
	cv	10.6%	NA	2.3%	12.3%

TABLE 5-8. SUMMARY OF QUENCH/SCRUBBER SYSTEM WATER pH DATA

Sample Stream	Run No.	Average pH	Standard Deviation	cv %	Range of Values
Quench	1-1	0.51	0.28	54.9%	0.06-0.88
Effluent	1-2	0.62	0.06	9.7%	0.52-0.74
	1-3	0.73	0.10	13.7%	0.66-1.02
	2-1	0.76	0.06	7.9%	0.66-0.85
	2-2	0.85	0.06	7.1%	0.78-1.01
	2-3	0.88	0.06	6.8%	0.81-1.03
	3-1	0.76	0.07	9.2%	0.68-0.83
	3-2	0.62	0.04	6.5%	0.56-0.71
	3-3	0.63	0.03	4.8%	0.58-0.70
Scrubber	1-1	1.59	0.38	23.9%	0.86-2.00
Effluent	1-2	1.82	0.03	1.6%	1.77-1.87
	1-3	1.80	0.09	5.0%	1.70-1.95
	2-1	2.31	0.16	6.9%	1.89-2.44
	2-2	2.33	0.04	1.7%	2.26-2.40
	2-3	2.31	0.04	1.7%	2.27-2.39
	3-1	1.73	0.06	3.5%	1.68-1.92
	3-2	1.58	0.05	3.2%	1.48-1.68
	3-3	1.59	0.04	2.5%	1.55-1.67
Utility	1-1	8.75	0.90	10.3%	6.80-9.27
Water	1-2	9.32	0.07	0.8%	9.20-9.43
	1-3	9.38	0.04	0.4%	9.33-9.44
	2-1	9.19	0.04	0.4%	9.11-9.25
	2-2	9.18	0.04	0.4%	9.11-9.24
	2-3	9.10	0.06	0.7%	9.02-9.17
	3-1	9.30	0.06	0.6%	9.19-9.39
	3-2	9.42	0.05	0.5%	9.27-9.48
	3-3	9.44	0.08	0.8%	9.23-9.51

6. QA/QC RESULTS

The purpose of a QA/QC program is to provide the environmental measurement systems employed in the trial burn with the procedures and documentation which demonstrate that the measurement has a defined accuracy and precision associated with it. Described herein will be QC results for maintaining instruments and equipment in a state of calibration (defines the accuracy or bias error), results for measuring/calculating the repeatability of a measurement (defines precision, or random error), results of maintaining a state of cleanliness (eliminates interferences or contamination), and the paper trail which documents that the methods were performed to instructions, calibrated, within method performance standards, traceable to NBS standard reference materials, audited, and samples were secure from tampering.

6.1 <u>Data Quality Objectives</u>

In the QA/QC Plan contained within the trial burn plan, data quality objectives for accuracy, precision, and completeness were outlined for the major measurement parameters. These objectives are sufficient to provide legally defensible data for RCRA permitting purposes.

6.2 Comparison of OC Results vs. Goals

Table 6-1 presents a summary of QC goals for precision, accuracy, and completeness from the trial burn plan. Highlighted in **bold numerals** on Table 6-1 are the actual QC results obtained as comparison against goals (in standard font). Excellent quality control was maintained for those QC parameters which were determined in this program.

6.3 <u>QA/QC Results Summary</u>

QA/QC results for the major measurements made in the trial burn are summarized in tabular form in this section. Additional QC results can be found in Appendix J -- QA/QC Results.

Table 6-2 presents the summarized results of matrix spiking of the POHCs (1,2-DCE and PCE) into the CAC Residue. A known amount of compound is spiked into an actual waste sample prior to sample extraction and analysis. The amount of analyte in the sample is preknown. Recoveries ranged from 80 to 102%, and duplicate analyses of the matrix spikes ranged from 8.2 to 20.2% difference, indicative of good analytical accuracy and precision for POHC analysis of the waste.

TABLE 6-1. QUALITY ASSURANCE OBJECTIVES FOR CAC INCINERATOR TRIAL BURN MEASUREMENT DATA

		Expected	l results,	8
Parameter	Method	Precision	Accuracy	Complete- ness
[NOTE: normal type	= QC goal; h	old type = a	ctual QC r	esult]
Liquid waste	EPA 8240			90
POHCs - EDC - Perc	(GC/MS)	±30 ^a ±8.2 ±30 ^a ±20.2	49-155 94-102 64-148 80-982	100
Aqueous samples	EPA 8240			90
POHCs - EDC - Perc	(GC/MS)	±30 ^a ±14.9 ±30 ^a +1.1	49-155 91-114 64-148 82-102	100
Stack gas POHC concen	EPA 0030			90
- EDC	(VOST)	±25 ^b NA	49 - 155 94-115	100
- Perc	EPA 8240 (GC/MS)	±25 ^b NA	64-148 102-108	
POHC concen	VOST audit gases	NA	50-150 Passed	
Velocity/flow	EPA 2	<u>+</u> 5 ^C	<u>+</u> 10 ^d	90 100
co_2 , o_2	EPA 3	±0.5 ^e	<u>+</u> 0.5 ^e	90 100

a Based on limits in EPA-600/8-84-002.

b Based on limits in the VOST Protocol, EPA-600/8-84-007.

C Based on method collaborative study, EPA-600/4-76-014.

d Estimated.

e As listed in EPA QA Handbook, Vol. III, EPA-600/4-77-027b. f Limit for analysis of control samples.

g Based on minimum performance specifications in 40 CFR 60, Appendix B, for CEMS.

TABLE 6-1. QUALITY ASSURANCE OBJECTIVES FOR CAC INCINERATOR TRIAL BURN MEASUREMENT DATA (Continued)

	Expected results, %						
Parameter	Method	Precision	Accuracy	Complete- ness			
[NOTE: normal type	= QC goal;	bold type = a	ctual QC r	esult]			
Stack gas Particulate	EPA 5	<u>±</u> 10 ^C not	listed	90 100			
HCl conc.	EPA 5; EPA 300.0	±30 ^a ±10.4	<u>+</u> 10 ^f + 5.5	90 100			
Oxygen conc.	EPA 3A	NA	<u>+</u> 1 ^g +0.3	90 96			
CO conc.	EPA 10	NA	<u>+</u> 10 ^g <u>+</u> 0.8	90 100			

a Based on limits in EPA-600/8-84-002.

TABLE 6-2. SUMMARY OF CAC RESIDUE MATRIX SPIKE QC RESULTS

Compound	Sample Conc ppb	+Spike Added ppb	Conc Found ppb	Recovery	Duplic Conc ppb	Duplic Recovery	Diff %
1,2-DCE	9	50	56	94.0%	60	102.0%	8.2%
PCE	51	50	91	80.0%	100	98.0%	20.2%

b Based on limits in the VOST Protocol, EPA-600/8-84-007.

Based on method collaborative study, EPA-600/4-76-014.

d Estimated.

e As listed in EPA QA Handbook, Vol. III, EPA-600/4-77-027b.

f Limit for analysis of control samples.

g Based on minimum performance specifications in 40 CFR 60, Appendix B, for CEMS.

Table 6-3 presents a summary of the surrogate recoveries of selected surrogate compounds spiked into the waste before extraction and analysis. Surrogates are organic compounds which are similar to the analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. In this program, deuterated 1,2-dichloroethane $(d_4-1,2-DCE)$, deuterated toleune $(d_8$ -toluene), and bromofluorobenzene (BFB) were spiked into the CAC Residue before extraction and analysis. Surrogate recoveries ranged from 79-113%, again indicative of good recovery of target analytes.

Table 6-4 presents a summary of the VOST sorbent surrogate recoveries. Surrogate compounds in methanolic solution are spiked using the flash evaporation technique into the VOST sorbent traps prior to thermal desorption and analysis, as part of the EPA Method 5040 protocol. Recoveries ranged from 93-122%, indicative of good analytical accuracy.

Table 6-5 presents the summary ofrecoveries from spiking sorbents with POHCs and selected PICs. The VOST sorbent, Tenax GC, performed well for recoveries of 1,2-dichloroethane and tetrachloroethene (POHCs) and for PICs such as carbon tetrachloride, trichloroethene, 1,1,1-trichloroethene, and chlorobenzene, with a range from 86-112%. The SemiVOST sorbent, XAD-2, did not perform well for tetrachloroethene with a recovery range of only 30-38%.

Table 6-6 presents a summary of the VOST analysis blank results. Field blanks were slightly contaminated with the city water pollutants; trip blanks showed slight contamination from acetone and methylene chloride, both used as sampling train cleanup and recovery solvents; and lab blanks showed slight contamination from acetone, a common lab air contaminant. None of the blank concentration levels presented any problems requiring blank correction.

Table 6-7 presents a summary of the stack gas chloride analysis QC results. Field blank solutions were relatively clean; percent recovery of matrix spikes ranged from 98-111%, indicative of good analytical accuracy; reference standards of chlorides performed well; and matrix spike precision ranged from 2-10% difference, indicative of good analytical precision.

QC results for other measurement parameters such as aqueous samples, GC/MS calibration and tuning, particulate blanks, TOX standard reference solution, semivolatile surrogate recoveries, and waste characterization parameter duplicates can be found in Appendix J.

Field equipment calibration results such as: 1) dry gas meters used in EPA Method 5, MM5, and VOST; 2) thermocouples; 3) digital temperature indicators; 4) electronic balance; and 5) barometer are shown in Appendix C.

TABLE 6-3. SUMMARY OF CAC RESIDUE QC SURROGATE RECOVERIES

		Surrogate Added	
	d4-1,2-DCE		BFB
Sample ID	*R 	%R 	%R
MQ-W-1-2-POHC	103	98	100
Analysis Blank	105	100	98
MQ-W-1-3-POHC	90	99	104
Method Blank	79	97	97
MQ-W-2-1-POHC	102	97	98
System Blank	97	96	97
MQ-W-2-2-POHC	89	99	99
MQ-W-2-3-POHC	91	99	100
System Blank	97	96	97
MQ-W-3-1-POHC	89	97	103
System Blank	80	97	98
MQ-W-3-2-POHC	106	103	102
MQ-W-3-3-POHC	113	106	106
System Blank	98	107	101

TABLE 6-4. SUMMARY OF VOST SORBENT QC SURROGATE RECOVERIES

	Su	rrogate Add		
	d4-DCE	d8-Toluen		
Sample ID	%R	%R	%R	
System Blank	106	108	109	
System Blank	95	99	96	
MQ5-VOST-1-1A	104	103	104	
MQ5-VOST-1-1B	104	107	108	
MQ5-VOST-1-1C	96	103	103	
MQ5-VOST-1-1D	94	104	95	
MQ5-VOST-1-2A	96	102	97	
MQ5-VOST-1-2C	98	105	106	
MQ5-VOST-1-3A	103	106	108	
MQ5-VOST-1-3B	97	106	107	
MO5-VOST-1-B	94	107	110	
MQ5-VOST-TB	103	103	103	
MQ5-VOST-1AU	97	105	105	
MQ5-VOST-2AU	102	110	106	
Lab Blank	100	101	101	
Lab Blank	96	96	100	
Audit	99	101	100	
MQ-VOST-2-1A	101	111	106	
MQ-VOST-2-1B	99	101	104	
MQ-VOST-2-1C	100	103	104	
MQ-VOST-2-2A	104	102	104	
MQ-VOST-2-2B	97	98	108	
MQ-VOST-2-2C	94	103	110	
MQ-VOST-2-FB	98	100	101	
MQ-VOST-2-3A	100	102	102	
MQ-VOST-2-3B	100	101	107	
MQ-VOST-2-3B	102	101	110	
MQ-VOST-3-1A	102	100	108	
MQ-VOST-3-1A MQ-VOST-3-1B	102	112	120	
MQ-VOST-3-1B MQ-VOST-3-1C	102	109	122	
MQ-VOST-3-FB	94	93	93	
MQ-VOST-3-FB	101	102	102	
MQ-VOST-3-2B	97	94	93	
MQ-VOST-3-2B	106	105	106	
MQ-VOST-3-2D	100	98	108	
MQ-VOST-3-2D MQ-VOST-3-3A	98	104	104	
MQ-VOST-3-3A MQ-VOST-3-3B		104	105	
	105 115	104	95	
MQ-VOST-3-3C	115	104	105	
MQ-VOST-TB-2	97			
Average	100.1	103.1	104.5	
cv	4.4%	4.1%	5.7%	

TABLE 6-5. SUMMARY OF QC REFERENCE STANDARD RECOVERIES FOR VOST AND SEMIVOST SORBENT TRAPS

Compound Spiked	Amt	added ng	Amt	found ng	Recovery %
VOST					
Carbon tetrachloride		250		252	100.8%
Trichloroethene		250		214	85.6%
Tetrachloroethene		250		254	101.6%
Chlorobenzene		250		246	98.4%
1,2-Dichloroethane		250		258	103.2%
1,1,1-Trichloroethane		250		240	96.0%
Carbon tetrachloride		250		281	112.4%
Trichloroethene		250		233	93.2%
Tetrachloroethene		250		270	108.0%
Chlorobenzene		250		255	102.0%
				Avg	100.1%
				CV	7.1%
SEMIVOST					
Tetrachloroethene		400		120	30.0%
Tetrachloroethene		400		140	35.0%
Tetrachloroethene		400		150	37.5%
				Avg CV	34.2%

TABLE 6-6. VOST ANALYSIS BLANK RESULTS

Compounds found	blar 	Field blanks, total ng ^a		Trip blanks ^b , total ng		Lab method blanks, total n			al ng
above detection limit	1-FB	2-FB	3-FB	TB-1	TB-2	1	2	3	4
Chloromethane	NDC	80	ND	ND	ND	ND		ND	ND
Methylene chloride	ND	ND	9	130	ND	ND	ND	ND	ND
Acetone	190	57	180	28	24	21	29		
Chloroform	24	84	ND	ND	ND			17	ND
Bromodichloromethane	10	ND	ND	ND	ND	ND ND	ND	ND	ND
Dibromochloromethane	5	ND	ND	ND	ND		ND	ND	ND
·lexanone	ND	29	ND	12	ND 17	ND	ND	ND	ND
Styrene	ND	ND	ND			ND	ND	42	ND
(ylenes	5	ND		ND	ND	ND	8	ND	ND
Chlorobenzene			ND	ND	ND	ND	7	ND	ND
	ND	6	ND	ND	ND	ND	ND	ND	ND
Benzene	ND 	ND	190	ND	ND	ND	ND	ND	ND

a Field blanks collected by assembling train at sampling site, leak checking, and recovering.

b Trip blanks - sorbents transported to field but not opened.

C ND - not detected (<3 ng)</pre>

TABLE 6-7. SUMMARY OF HCl AND Cl₂ QUALITY CONTROL RESULTS

Sample ID	Type of Sample	Cl added mg/L	Chloride mg/L	Recovery %	Diff %
Blank Na2CO3	Blank		0.23	NA	NA
Blank H2SO4 Blank Na2CO3	Blank Blank		4.33 0.23	NA NA	NA NA
MQ-S-CL-2-2	Method Spike		2.02	101.0	
MQ-S-CL-2-2 MQ-S-CL-2-3	MS Duplicate Method Spike		2.22 1.96	111.0 97.8	10.4%
MQ-S-CL-2-3	MS Duplicate	2.0	1.97	98.3	1.7%
SRS 1	Ref Std	4.0	3.99	99.8%	NA
SRS 2	Ref Std	4.0	4.03	100.8%	NA
SRS 3	Ref Std	4.0	4.13	103.3%	NA
SRS 4	Ref Std	4.0	4.22	105.5%	NA

Table 6-8 presents the summary of the stack gas oxygen CEM relative accuracy test, which indicates acceptable performance during the trial burn.

TABLE 6-8. RESULTS OF O₂ CEM RELATIVE ACCURACY TESTS^a

		0 ₂ concent	ration, %	,		
Date (1989) Test No.		Determined by Orsat	Determined by CEM	Difference, % 0 ₂ b		
1/31	1-1	10.5	10.8	0.3		
2/1	1-2	9.9	10.3	0.4		
2/2	1-3	10.0	10.1	0.1		
2/10	2-1	10.8	11.0	0.2		
2/11	2-2	10.8	11.2	0.4		
2/14	3-1	9.8	9.5	0.3		
2/16	3-2	10.0	10.3	0.3		
2/17	3-3	9.2	9.4	0.2		

^a Conducted as follows: After completion of Orsat analysis, bag sample was attached to calibration line and sampled through entire CEM system until a steady reading was obtained.

b Quality assurance objective: $\pm 0.5\%$ O₂ based on limit in EPA Quality Assurance Handbook, Vol. III, EPA 600/4-77-027b.

Tables 6-9 through 6-11 present QC results of the oxygen CEM pre- and post-test calibration data for Test Conditions 1 through 3, respectively. Tables 6-12 through 6-14 present QC results of the carbon monoxide CEM pre- and post-test calibration data for Test Conditions 1 through 3, respectively.

TABLE 6-9. OXYGEN CEM CALIBRATION DATA, TRIAL BURN CONDITION 1

Standard concen-	concen-		Analyzer response, % of scale		12		
Test No.	tration, %	Pretest	Posttest	Drift, % of span	Linear regression equation	Correlation coefficient	
1-1	14.96 10.10 4.01 0.0	68.0 47.2 21.6 4.4	68.5 46.1 19.0 4.5	0.7 1.6 3.8 ^a 0.2	Cor.c. $\% = \frac{CD - 4.477}{4.240}$	0.99999	
1-2	14.96 10.10 4.01 0.0	67.6 45.2 19.0 4.4	67.5 44.1 18.5 4.4	0.1 1.6 0.7 0.0	Conc. $\% = \frac{CD - 3.218}{4.243}$	0.99904	
1-3	14.96 10.10 4.01 0.0	65.3 44.5 20.2 4.6	64.2 43.7 20.0 4.4	1.7 1.2 0.3 0.3	Conc. $\% = \frac{CD - 4.211}{4.0508}$	0.99980	
HC1/C1 Tests 1-1 and 1-2	14.96 10.10 4.01 0.0	64.0 45.8 19.9 4.3	62.3 44.9 19.2 4.5	2.7 ^a 1.4 1.1 0.3	Conc. $\% = \frac{CD - 4.237}{4.027}$	0.99970	

a Excess limit for Method 3A, ± 2% of span.

TABLE 6-10. OXYGEN CEM CALIBRATION DATA, TRIAL BURN CONDITION 2

Standard concen-	Analyzer response, % of scale		D 16.			
Test No.	tration, %	Pretest	Posttest	Drift, % of span	Linear regression equation	Correlation coefficient
2-1	14.96	65.5	65.5	0.0		
	10.10	45.1	44.5	0.9	Comp	0.0000
	4.01	20.3	20.1	0.3	Conc., $\% = \frac{\text{CD} - 4.209}{4.079}$	0.99993
	0.00	4.5	4.6	0.2		
2-2	14.96	64.6	65.0	0.6		· · · · · · · · · · · · · · · · · · ·
	10.10	44.4	45.0	0.9	c cD - 4.628	
	4.01	20.9	20.4	0.8	Conc., $\% = \frac{\text{CD} - 4.628}{3.990}$	0.99990
	0.00	4.6	4.8	0.3	3.330	
2-3	14.96	64.5	68.0	5 1a	CD 4 274	
	10.10	44.5	50.0	5.4 ^a 8.5 ^a 2.2 ^a		
4.01	20.4	21.8	2.3a	Conc., $\% = \frac{\text{CD} - 4.374}{4.004}$	0.99996	
	0.00	4.5	24.5	31.0 ^a	4.004	

 $^{^{\}rm a}$ Exceeds limits of Method 3A, ± 2 % of span drift; last 45 minutes of test period voided because of calibration error.

TABLE 6-11. OXYGEN CEM CALIBRATION DATA, TRIAL BURN CONDITION 3

Standard concen-	Analyzer % of	Analyzer response, % of scale				
Test No.	tration, %	Pretest	Posttest	Drift, % of span	linear regression equation	Correlation coefficient
3-1	14.96	64.0	63.5	0.3		
	10.10	44.5	44.2	0.0	Cama w _ CD - 3.794	0 00070
	4.01	19.0	20.3	0.1	Conc. $\% = \frac{\text{CD} - 3.794}{4.015}$	0.99970
	0.00	4.4	4.5	0.0		
3-2	14.96	63.5	63.0	0.8		
	10.10	43.2	44.9	0.8 2.7 ^a	c cD - 3.935	0.0076
	4.01	19.4	19.0	0.6	Conc. $\% = \frac{CD - 3.935}{3.948}$	0.99976
	0.00	4.4	4.5	0.2	0.7.0	
3-3	14.96	64.2	61.0	5.0 ^a 4.0 ^a 2.2 ^a	CD - 4.264	
	10.10	44.6	42.0	4.0 ^a		0.0000
	4.01	20.0	18.6	2.2 ^a	Conc. $\% = \frac{CD - 4.264}{3.999}$	0.99996
	0.00	4.5	4.5	0.0		

^a Drift exceeds limit of Method 3A, \pm 2% of span.

TABLE 6-12. CARBON MONOXIDE CEM CALIBRATION DATA, TRIAL BURN CONDITION 1

Standard concentration, Test No. ppm	Analyzer % of	response, scale	Drift,			
	· ·	Pretest	Posttest	brift, % of span	Linear regression equation	Correlation coefficient
1-1	252.6	90.4	90.4	0.0		
	99.7	40.0	40.5	0.6	CD - 5.742	0.0004
	49.8	22.9	23.2	0.3	$ppm = \frac{CD - 5.742}{0.337}$	0.99984
	0.0	5.0	5.9	1.0		
1-2	252.6	88.1	88.5	0.5	**************************************	
	99.7	39.5	39.0	0.6	$ppm = \frac{CD - 6.250}{0.326}$	
	49.8	22.8	22.2	0.7		0.99981
	0.0	5.5	5.1	0.5		
1-3	252.6	90.9	91.4	0.6		
	99.7	40.4	40.9	0.6	CD - 6.404	
	49.8	23.2	23.3	0.1	$ppm = \frac{CD - 6.404}{0.335}$	0.99993
	0.0	6.0	5.7	0.3	3.000	
HC1/C1 ₂	252.6	92.5	92.3	0.2		
tests	99.7	39.2	92.3 41.2	0.2	$ppm = \frac{CD - 5.247}{0.345}$	
1-1, 1-2	49.8	23.1	24.0	2.2 1.0		0.99992
1-1, 1-2	0.0	5.0	6.3		0.345	
	0.0	J.U	0.3	1.4		

TABLE 6-13. CARBON MONOXIDE CEM CALIBRATION DATA, TRIAL BURN CONDITION 2

Standard concen-	Analyzer % of s	Analyzer response, % of scale					
Test No.	tration, ppm	Pretest	Posttest	Drift, % of span	Linear regression equation	Correlation coefficient	
2-1	252.6	88.8	87.5	1.5	······································		
	99.7	39.0	38.0	1.1	CD - 5.604	_	
	49.8	22.2	21.0	1.4	$ppm = \frac{CD - 5.604}{0.330}$	0.99994	
	0.0	5.2	4.5	0.8			
2-?	252.6	91.0	89.8	1.3			
	99.7	40.2	39.9	0.3	CD - 5.767		
	49.8	22.7	22.5	0.2	$ppm = \frac{CD - 5.767}{0.339}$	0.99991	
	0.0	5.3	5.1	0.2	0.333		
2-3	252.6	91.0	90.5	0.5	$ppm = \frac{CD - 5.672}{0.339}$		
	99.7	40.2	39.0	1.3			
	49.8	23.0	22.5	0.5		0.99983	
	0.0	4.9	5.6	0.8	0.005		

TABLE 6-14. CARBON MONOXIDE CEM CALIBRATION DATA, TRIAL BURN CONDITION 3

Standard concen-	Analyzer % of s	Analyzer response, % of scale					
Test No.	tration, ppm	Pretest	Posttest	Drift, % of span	Linear regression equation	Correlation coefficient	
3-1	252.6	90.0	90.2	0.2			
	99.7	37.4	40.2	3.1	CD - 4.963		
	49.8	22.0	23.0	1.1	$ppm = \frac{CD - 4.963}{0.335}$	0.99983	
	0.0	5.3	5.6	0.3			
3-2	252.6	90.0	89.6	0.4			
	99.7	40.0	36.5	3.9	CD - 5.972		
	49.8	22.9	21.9	1.1	$ppm = \frac{CD - 5.972}{0.334}$	0.99985	
	0.0	5.3	4.9	0.4	0.001		
3-3	252.6	90.8	90.9	0.1	$ppm = \frac{CD - 5.646}{0.338}$		
	99.7	40.0	40.3	0.3			
	49.8	22.7	23.0	0.3		0.99990	
0.0	0.0	5.1	5.1	0.0	3.335		